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PRODUCTION CARBURIZING

By Ernest F. Davis

Abstract

The initial part of this paper is devoted to the antiquity of carburizing showing that cementation as exemplified in the Harveyizing process was an outgrowth of methods practiced by the ancients. Sustaining evidence from metallographic tests of Egyptian implements and reports of medieval practices by various writers are quoted to demonstrate that early workers in iron utilized this means of imparting carbon to steel and furthermore it was in common use during the Middle Ages.

The less refined methods in vogue when the automobile industry was young are cited. The article then shows chronologically the gradual improvements in the practice which has affected the quality and costs of the process such as the change from coal-fired to oil and gas-fired furnaces, the advent of the pyrometer, the elimination of manual control, the rise of scientific supervision, the development of carburizing compounds, the influence of the heat resisting alloy and the factors which brought about the mechanized furnace. The construction and operation of some of the modern installations are given in detail. Also included are items often ignored which affect carburizing costs.

CARBURIZING or "carbonizing" as it was called until about fifteen years ago, is probably one of the oldest of the ferrous arts. It is unlikely that such a simple process of packing iron with carbonaceous material in some kind of a sealed container would have remained unknown very long after the more difficult operation of reducing iron ore into useful metal had been accomplished. Although still termed "cementation" in Europe our present box carburizing is not an outgrowth of the well known "cementation process" as many writers have indicated but was one of the means of imparting carbon to the hammered spongy iron made long before the Christian era. Actually cementation as exemplified in the Harveyizing and earlier

A paper presented as part of the Symposium on Carburizing presented at the Nineteenth Annual Convention of the American Society for Metals held in Atlantic City, October 18 to 22, 1937. The author, Ernest F. Davis, is chief metallurgist, Warner Gear Co., Muncie, Indiana.

methods of making blister steel are all improved adaptations of primitive methods of carburizing.

Metallographic examination of a sickle, knife, chisel, hoe and axehead from Egyptian tombs from 1200 to 200 B.C. reported by Sir H. C. Carpenter and J. M. Robertson* clearly demonstrated that at least some of these had been made into useful tools by means of carburizing. In the examination of a knife for example they state in part:

"From the edge toward the back the carbon content decreases slowly . . . From the point toward the back of the knife the carbon content decreased rapidly and at this point was nearly carbonless . . . The authors conclude that the knife was made by welding two pieces of metal together and was then carburized to make the content at the edge at about 0.80 per cent carbon."

G. E. Thackery† states that the examination of arms, tools, spears and plates of armor from India of quite ancient origin have shown that many of these were apparently carburized.

We must consider that the making of useful iron and steel articles for many centuries was performed in small shops scattered all over Europe and Asia with rudely constructed furnaces and that some of their processes were unbelievably slow and tedious. Not until the 16th century was the iron and steel industry centralized and commercialized and then it often developed into a royal monopoly. The early workers in iron and steel did not know the part carbon played in conferring upon iron the ability to harden but attributed it to some magical ingredient, celestial agency or phlogistic potency, hence we find mentioned in the compositions such items as black ox blood, "toade oyle," fen moss, viper flesh, mummy powder and many other concoctions equally as ridiculous. However fantastic in formulae, we find all of these carburizing agents were essentially of animal and vegetable carbons containing bone, lamp black and charcoal; and nitrogenous bodies such as ground horn, hoof parings, chicken and animal dung, etc. The word "coal" which often appears in these ancient formulae refers to charcoal and not anthracite or bituminous varieties.

The containers first employed were no doubt of clay. The iron was probably placed in bags containing the carburizing powder and

*The Metallography of Some Ancient Egyptian Implements, *Journal*, Iron and Steel Institute, Vol. CXXI, p. 433.

†G. E. Thackery, TRANSACTIONS, American Society for Steel Treating, October, 1924, p. 147.

the bag smeared with clay mixed with salt which was then dried and baked. The mass so prepared was then placed in a furnace similar to the Catalan or Belgian forge and heated for several days. This is a logical assumption for the forming of vessels from clay is one of man's most primitive arts.

In the early part of the 17th century John Hayden* hardened whole bars of iron by heating them from 3 to 7 days in "Coffins of Amblecot Clay" each coffin holding about one-half ton of iron laid in coal (charcoal).

William Ellyot and Mathias Meysey* were granted patents in England in 1614 as follows:

"Which converting of iron into steele is performed by meanes of a reverboratorie furnace with pottes luted and closed to be put therein contayning in them certain quantities of iron with other sybstances, mixtures and ingredients being in the same furnace brought to a certain proporcion of heat doth make or convert the same iron into steele which steele with other heates temperature and hammering to be afterwards given doth make yt good and fill for the uses before mencioned."

Unquestionably much useful steel was made during the Middle Ages by carburizing processes. It is said that the carriage springs used in the 17th century about the time Charles II was king of England were manufactured from wrought iron which was carburized in containers, hardened and then tempered sufficiently to eliminate brittleness.

Before the stimulation given to case hardening by the bicycle industry the box method had declined although the cementation process flourished in the industries making armor plate and cutlery. About this period crucible steel, then called cast steel, was growing in popularity and was less expensive with the result that the demand for "artificial steel" was extremely small. However it never reached the point of obsolescence because the manufacturers of gun mechanisms and sewing machines employed it for a number of years and some gears were also carburized. With the advent of the bicycle industry bearings and cones were carburized. Copper plating as a carburizing resist was used in 1895.

Rhead's Metallurgy† published in 1902 describes case hardening as follows:

*The History of the Cementation Process in Steel Manufacture, *Journal*, Iron and Steel Institute, Vol. CXXI, p. 463.

†Rhead's Metallurgy, 1902, p. 147.

"The surface of wrought iron and mild steel articles are often superficially hardened by packing them in iron boxes with parings of hoofs and horns, leather scrap, bone dust and charcoal; and heating to full redness. The depth of hardening depends upon the length of time they are heated."

An early edition of Kent's Mechanical Engineers Handbook* gives the following description:

"Some makers carburize the surface of gears . . . They are packed in cast iron boxes with a mixture of bone and powdered charcoal and heated for four hours at nearly the melting point of the boxes, then cooled slowly in the boxes. They are then taken out, heated to 1400 degrees Fahr. for four hours to break up the coarse grain produced by the carbonizing temperature."

The interesting thing in the last two references is that the ingredients used in the early days of the bicycle and automobile industries were exactly the same as those described by writers from the 14th to the 18th centuries and furthermore was almost as crudely done forty years ago as it was practiced several centuries before. In fact a carburizing compound sold thirty years ago contained particles of horn, leather strips, potassium nitrate, bone and wood charcoal, all ingredients which entered into the composition of ancient carburizers. These same substances plus potassium cyanide and the prussiates constituted the "secret" surface hardening powder used by blacksmiths for many years by sprinkling on the surface of wrought iron and machinery steel after heating in the forge.

Although natural gas and producer gas were the standard furnace fuels when available yet most of the early production carburizing furnaces in operation forty years ago were coke or coal-fired and provided with fire boxes, grate bars, ash pits and flues for conducting the hot gases into the carburizing chamber and utilizing the reverberatory principle of reflecting the heat downward from an arched ceiling. A typical design is shown in Fig. 1. To provide the necessary draft each furnace was equipped with individual chimneys. In one plant in 1906 quite an advance in furnace design was accomplished when eight of these coal fired furnaces were grouped into one unit and the flues connected to a tall central stack.

The standard carburizing temperature at that time, and evidently copied from European cementation practice, was 900 to 1000 degrees Cent., which is 1650 to 1830 degrees Fahr., as if this difference did

*Kent's Mechanical Engineers Handbook, 1910, p. 406.

not make any variable in case depth and the condition of the product. But the actual temperature used was more or less mythical for furnaces were hand fired, there was no pyrometric control and the heat attained was dependent upon the observations of the furnace attendant or fireman through a peep hole in the door and who held the temperature to a "dark orange."

The men in charge of these early heat treating departments were usually exalted blacksmiths who at that time were the only ones with any knowledge of the heat treatment of steel. This statement is

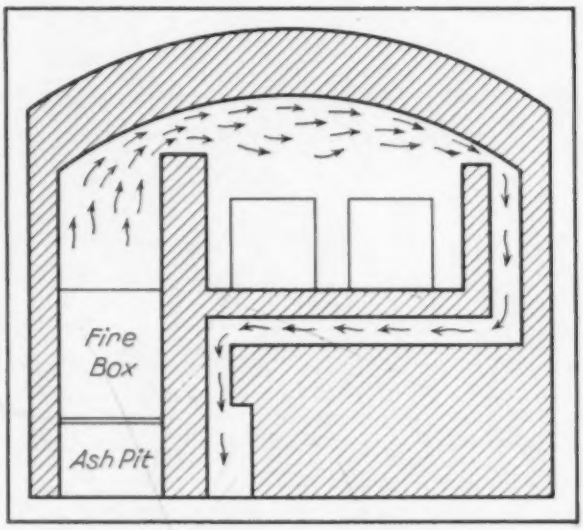


Fig. 1—Sketch of Coal Fired Furnace.

made without any intent of ridicule for these men were practical workers in iron and steel and were often very clever in spite of the meager scientific knowledge at their disposal and who had only the precedence of their forbears in the art and the facts gained by their own experiences. When the pyrometer was first introduced about 1905 most of the older men refused to use them and resented their advent as a reflection upon their ability to accurately determine temperature by ocular observation.

The cost to carburize during this period was from ten to twenty cents per pound and the quality of work usually poor.

As the automobile grew in importance and the demand for gasoline increased with the parallel opening of numerous oil fields, the oil industry acquired an accumulation of inferior crude oils and oil residue from which the lighter and more marketable distillates had been removed. These oils tested from 30 to 40 Baumé and made

excellent furnace fuels. This oil was offered to industry initially at a price of approximately 2 cents per gallon under the name of "petroleum" which was later designated as "fuel oil." Although oil even at this price was higher than coal the saving in labor by the elimination of firemen more than compensated the difference.

At first it was fired in the same fuel boxes as had been used for coal but it was soon learned it could be more efficiently burned by

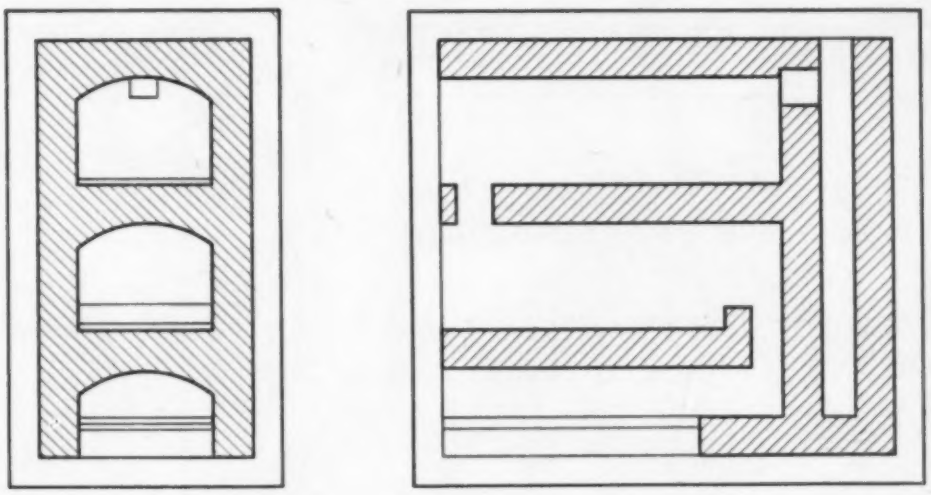


Fig. 2—Early Type Two Chamber Coal Fired Furnace.

introducing it under pressure atomized with compressed air or dry steam instead of utilizing natural draft. Burners were then devised to spray the oil into the combustion chamber. This rapidly brought a change in the design of carburizing furnaces for no longer were the high stacks, grate bars and ash pits essential but required combustion chambers of ample size to completely consume the fuel. The familiar square or rectangular box carburizing furnace then came into being and is still in service in many plants at the present time. In Europe few plants have developed beyond this for we find box furnace practice used almost entirely abroad. This is not due to lack of modernization, but automobile plants are smaller and the fuel situation is different than in the United States for there is no natural gas in the industrial countries such as France, Germany and England.

The box furnace has been fired many ways. Some were direct fired with the flame entering above the box level but this type was soon discarded due to the cold floor or hearth. Some were overfired with a combustion chamber between a double arch in the roof having openings in the brickwork of the lower arch to direct the flame down-

ward onto the boxes. These had the same fault of the direct fired and the arch repairs were quite frequent. However most of the furnaces were underfired with the burners placed on the sides, front or rear. This insured a hot hearth. Flues conducted the heat up along the side and rear walls into the carburizing chamber. Sometimes for economy these furnaces were constructed in pairs and fired from the outer sides.

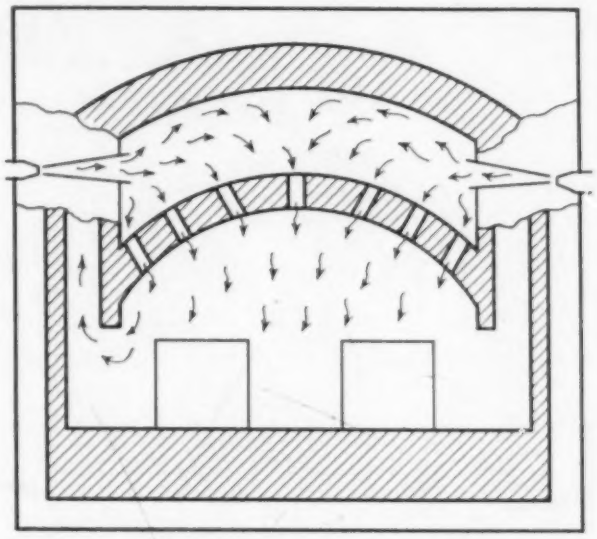


Fig. 3—Over-Fired Oil or Gas Furnace.

One of the largest installations in the United States from 1914 to 1923 was fired with burners beneath the furnace. These were housed in a building about 800 feet long and consisted of two rows of 24 furnaces or a total of 48 furnaces. Each furnace was fired with a single burner of large size which impinged on a fire clay spreading disk in the center of the furnace bottom. The furnace piers supporting the hearth were radiant from the burner thus permitting equal distribution of the flame. Below each row of furnaces were long tunnels extending the length of the building for lighting the burners, replacing the baffle disks and making furnace repairs.

All the pyrometer control leads were connected to a master control station and each burner had an electrical device for increasing or decreasing the fuel supply which was wired to a control panel. By a switch dialing arrangement the furnaces in operation were checked at definite time intervals with the master indicator, and by means of buttons the supply of fuel could be increased or lessened in accordance to the temperature shown. By this means the cycle of

each heat was maintained by manual control. Signal lights acquainted the operators when the furnaces were ready to be unloaded.

For convenience of operation nearly all plants built their furnaces in rows. Some had charging cars on tracks before the furnaces and having their top plates on a level with the furnace hearth. Some employed elevating trucks, often provided with long arms for charging the pots into the furnace and withdrawing them at the completion

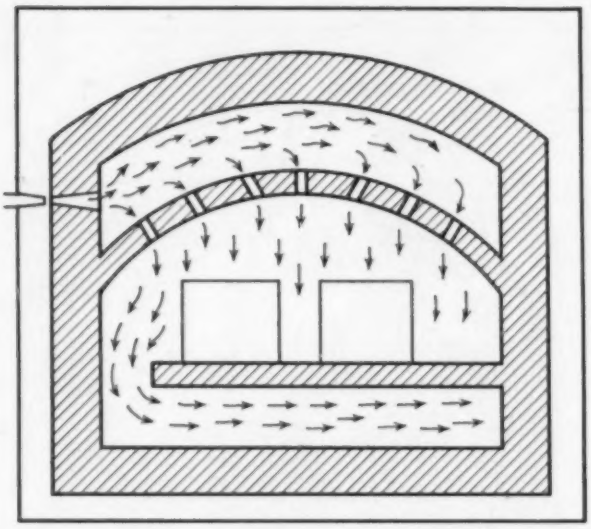


Fig. 4—Over and Underfired Carburizing Furnace.

of the heat. Furnaces utilizing these elevating trucks had short piers above the level of the furnace and the boxes were laid straddling these piers so that the charging arms could be lowered and withdrawn after depositing the boxes, and also to facilitate the removal of the charge at the finish of the heat. In a number of heat treating departments the furnaces were sunk in pits or designed so that the hearth level would be on the floor level to eliminate the arduous lifting of boxes in and out of the furnace. However, a majority of the smaller plants, and even some of the larger ones, handled their boxes by pure muscular effort with dollies on wheels or other charging tools. It is obvious that the firms equipped with the most efficient labor saving devices showed better cost figures than those where this work was laboriously and awkwardly performed.

The period from 1910 to 1920 was one during which much carburizing was done. The mild steels of the carburizing grade were predominate in motor car construction. Not only nearly all gears but nearly all the stressed and wearing parts, even including crankshafts. During this decade we find the metallurgist gradually replacing the

blacksmith in domination of heat treating operations, being recruited principally from the chemical profession. The thermocouple also replaced the human eye and the recording pyrometer, giving the complete history of each carburizing heat.

The practical man however did not yield easily to the scientifically trained investigator. In 1919 T. J. Sellick,* referring to the timidity of practical men to comment at meetings in the discussion of carburizing problems, said:

"Don't be afraid of the metallurgist. The chances are that in your experiences you meet problems that if presented to him would give him much food for profound thought."

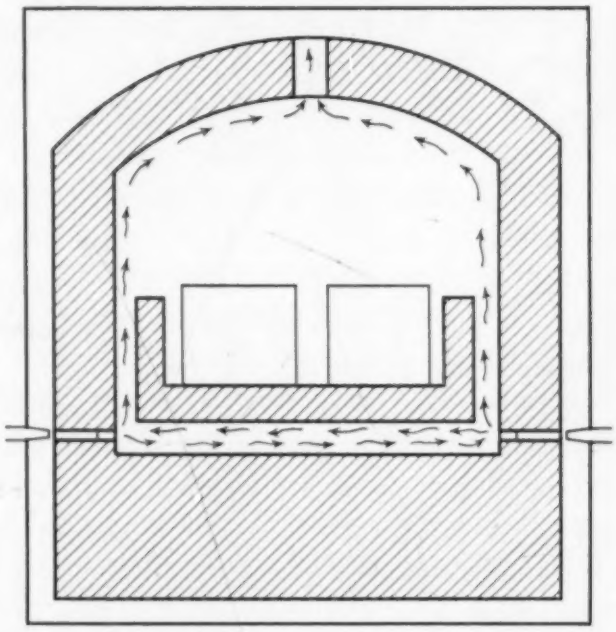


Fig. 5—Standard Underfired Carburizing Furnace.

If one scans the heat treating literature published during this period we find much of it devoted to the defects of carburizing practice. Softness of carburized work was common. Brittleness of steel after carburizing caused much concern. Cracking, checking and exfoliation were serious troubles. Pitting on the surface, adhesions and fusions of foreign matter, irregular depth of case, uneven hardness and many other items could be listed. The work of McQuaid and Ehn published in 1922 was an outgrowth of their investigation of the variables in case hardened work. This valuable contribution to ferrous metallurgy was immediately seized upon and the steel

*T. J. Sellick, *Journal*, American Society for Heat Treating, Vol. 2, 1919.

maker immediately became the goat for all the defects of case hardened work. Most of the prejudice which exists today against the process of carburizing and case hardened products hearkens back to the crudities of early practices.

For many years most of the work carburized was by the medium of raw ground bone, and in many of the early text books written prior to 1910 would frequently appear the statement that the brittleness of case hardened work was due to the phosphorus of the bone. This assumed that the stable phosphates of the bone would jump into the steel. This belief was prevalent a number of years ago but reveals the general opinion that the carbon as well as other elements were absorbed by contact with the heated steel.

Bone was extremely heavy, weighing from 55 to 61 pounds per cubic foot, depending upon its mesh, and contained from 45 to 50 per cent inert matter. Bone cost from $45.00 to $75.00 per ton, and due to the fact that it could be used but once was one of the most expensive of the early carburizers and contributed much toward the high costs to case harden in the early days.

This was superseded by compounds composed largely of charred leather with bone, and bone with charcoal. One said to have been originally exhausted bone from oil filters and comprised of heavy oil and finely ground bone, was called hydrocarbonated bone black. This latter compound was very popular in the bearing industry and was in use until very recent years. Leather and charcoal composed another mixture. A patented compound consisted of charred leather soaked in caustic soda and another sodium compound which was afterwards heated from 1400 to 1600 degrees Fahr. (760-870 degrees Cent.). Sawdust mixed with soda ash constituted another. A deviation from this was wood chips soaked in soda ash and sodium silicate (water glass) and then charred.

After 1910 the gradual trend was from the leather compounds and toward the energized or chemical type. These were mixtures of charcoal, Solvay coke, retort carbon with energizers and catalysts such as iron oxide, sodium phosphate and acetates, and the carbonates of calcium, barium, lithium, sodium and strontium. One popular brand had the charcoal soaked in a strong solution of soda ash which was afterwards dried and mixed with coke of the same mesh. Another type was impregnated with energizers on the charcoal grains. The grains were covered with pitch and the mass charred. Others were coal or other carbons powdered, mixed with energizers and

impregnated with a tacky binder. By mechanical shakers the grains were formed into dense pellets the size of a pea and coked. Oil was also used as a binder in several compounds to affix the energizing powder to the grains.

At one time there was no less than twenty types of case hardening compound marketed. Some of the most important ones were Petinos, Bulldog, Pearlite, Woodside, Carbo, Atlas, Bohnite, Stecco, Blaiches Modern, Quicklight, Standard, and others. These various compounds weighed from 11 to 61 pounds per cubic foot, being the lightest in those essentially of charcoal grains and heaviest in the finely ground bone. Most of the compounds sold under trade names weighed from 22 to 32 pounds per cubic foot.

The replacement of bone by the chemical or energized carburizers was a big step toward the reduction of case hardening costs. Compounds were developed which were not only of greater volume per ton but which could be repeatedly used without any marked deterioration in their carburizing potency. Instead of a 50-50 per cent mixture, which was common practice when bone was commonly used, a mixture of 80 per cent old and 20 per cent new became the mixing formula. In present day practice compound is re-used an indefinite number of times being merely regenerated by replacing the volume lost by burning and dusting.

Today the mixture is more or less standardized of which the following is a typical specification:

Hardwood Charcoal	53 to 55 per cent	(Hickory, willow, etc.)
Solvay Coke	30 to 32 per cent	
Sodium carbonate	2 to 3 per cent	(Soda ash)
Barium carbonate	10 to 12 per cent	(Precipitated)
Calcium carbonate	3 to 4 per cent	(Whiting)

Some firms do not like the coke which is essentially a filler and is mainly useful in reducing shrinkage. In place of the above they specify a treated charcoal containing 82 to 85 per cent charcoal and the remainder energizer in about the proportions shown above. Some specifications have eliminated the sodium carbonate due to a belief it is destructive to alloy boxes, the cause of surface fusions and contributary to the creep of copper when used as a carburizing resist.

Boxes were of cast iron, cast steel, annealed and unannealed malleable iron, and occasionally pressed steel. Elongated parts such as camshafts were carburized in pipes and tubing. Cast iron was the cheapest and the metal most commonly used. It gave about 250 hours average life before failure, which meant that they were fre-

quently cracking during the progress of a heat and permitting the escape of the carburizing gases. Good cast steel averaged about 450 heat hours and white iron and malleable about 350 to 400. White iron frequently cracked during handling.

The boxes were packed in the conventional manner, a lid was placed on top and the edges luted with mud or fire clay. The box furnace previously brought to temperature was loaded with as many boxes as it would hold. The advent of this cold charge of course chilled the furnace and the burners were then forced to bring it up to temperature as quickly as possible. The period of carburizing was from 6 to 14 hours allowing 4 to 6 hours to bring the box up to heat. These figures varied, depending upon the size of the boxes, the type of furnace and the depth of case desired. During this period the burners had to be adjusted continually by hand to keep the furnaces within reasonable limits, which the operator usually did unless he forgot, grew careless or was otherwise engaged. After a standardized interval one of the boxes was pulled out to the forehearth, a test pin was pulled out and this quenched and broken and if the case depth as measured with a scale was correct the entire heat was pulled by forked tools on rollers or other means previously described, and the boxes laid out to cool or dumped upon coarse meshed screens which would allow the carburizing compound to sift through, and the parts then quenched in oil or water.

It is obvious that the chilling effect of filling the furnace with cold boxes would upset the heat balance. Those in more favored localities in the furnace chamber would heat up faster than others. Actual tests made with thermocouples placed in the center of each box in a carburizing heat revealed some interesting facts. A difference of 250 degrees Fahr. was noted in the first hour in the heating rate of different boxes. By the time the first box was up to 1700 degrees Fahr. temperature the lowest box was 1525 degrees Fahr. One and one-half hours additional time was required before the last box was up to the indicated temperature of the furnace. One box showed 35 degrees Fahr. higher than the furnace itself and consistently held this through the entire cycle. In another test of five boxes with a desired case depth of 0.060-inch a variation of 0.016-inch occurred between the product of the different boxes. Many writers on carburizing practice have verified the existance of these variables in the average production carburizing operations.*

*T. J. Sellick, *Journal*, American Steel Treating Society, Vol. 2, 1920, p. 206.

The preceding is a cross section of case hardening practice as it was performed in American industry for over 30 years, and in many places it is yet actually the same where box furnace procedure is still in vogue. A case depth variation of 0.010-inch was regarded as the closest limit possible by box furnace practice.

The cost to carburize in plants from 1910 to 1924 was improved over earlier practice and varied from 3 to 10 cents per pound due to the various factors mentioned. Often it was nearer the 10-cents than the 3-cents cost. In one large plant during 1921 a cost of 3.8 cents per pound was obtained, based upon 12,000 pounds of miscellaneous work. During this same year much of the equipment of the plant was idle and it comprised a considerable investment. The costs for the year of 1921 to carburize was 8.4 cents per pound.*

In another large plant† the cost to carburize 1000 pounds of steel in 1924, allocated as shown, was 3.2 cents per pound.

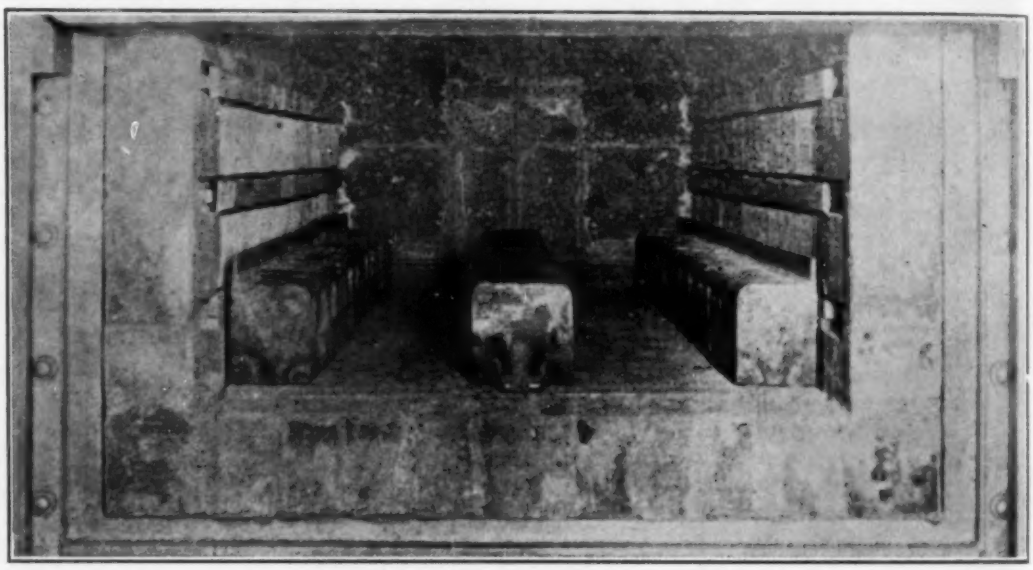
Boxes and lids	$8.65	(Cast iron)
Fuel	8.63	(Natural gas @ 51¢)
Carburizing compound	3.56	
Luting clay	0.15	
Furnace repairs	1.19	
Furnace depreciation	1.66	
Labor	5.52	
Overhead charges	2.76	(50% of labor)

One of the first steps toward improving uniformity was automatic fuel control. This was preceded by automatic signalling pyrometers utilizing colored lights. Usually three lights were employed, a green light which would burn when the temperature was below the required limit and a red light when the temperature was too high. When the furnace was operating between a limit usually set at 25 degrees a white light burned. Oil and gas proportioning valves were employed for many years but they were seldom used in carburizing furnaces until about 1920. The introduction of the electric box furnace for carburizing about this year simplified the problem, for electric heat could be turned on and off by simple contacts and relays and functioned with great sensitivity with the pyrometer indicators. Following this several excellent oil and gas controls were developed and the uncertainty of manual firing was eliminated.

Although heat resisting alloys were known from 1906, their initial application was solely for thermo-electrical purposes such as the heat units employed in the first Hoskins furnaces. The initial cost of this

*Willys Morrow Co., Elmira, N. Y.
†Warner Gear Co., Muncie, Indiana.

alloy was about $5.00 per pound. In 1918 Henderson patented the nickel-chromium alloys for carburizing boxes. The adoption of this alloy increased the life of boxes from about 250 hours for the cast iron to 8000 to 10,000 hours with the alloy. It was demonstrated by C. M. Campbell* in 1922 an alloy box at $1.25 per pound was more economical than a cast steel box, although the former cost $56.25 more than the latter. At first the high initial cost discouraged their adoption but their value was soon apparent. These alloys had much



Piers in Furnace for Loading and Unloading with Elevating Trucks.

higher strength than the cast iron and cast steel at elevated temperatures and could stand more handling without distortion with furnace tongs and gripping tools. In addition this lessened the frequency of cracked boxes and thus improved the quality of carburized work by reducing lean carburizing, scaling and soft work attributed to loss of gas. Another important contribution of this alloy was the possibility of placing mechanical equipment, roller rails and metallic hearths within the furnace itself and still maintain the underfiring principle. Roller rails, for example, reduced the friction of pushing work through the furnace chamber thus enabling longer furnaces to be built and paved the way for the modern designs of mechanized furnaces.

However, the first important step toward improving the quality and eliminating the uncertainty of box furnace practice was the rotary hearth furnace, so-called because it was designed with a

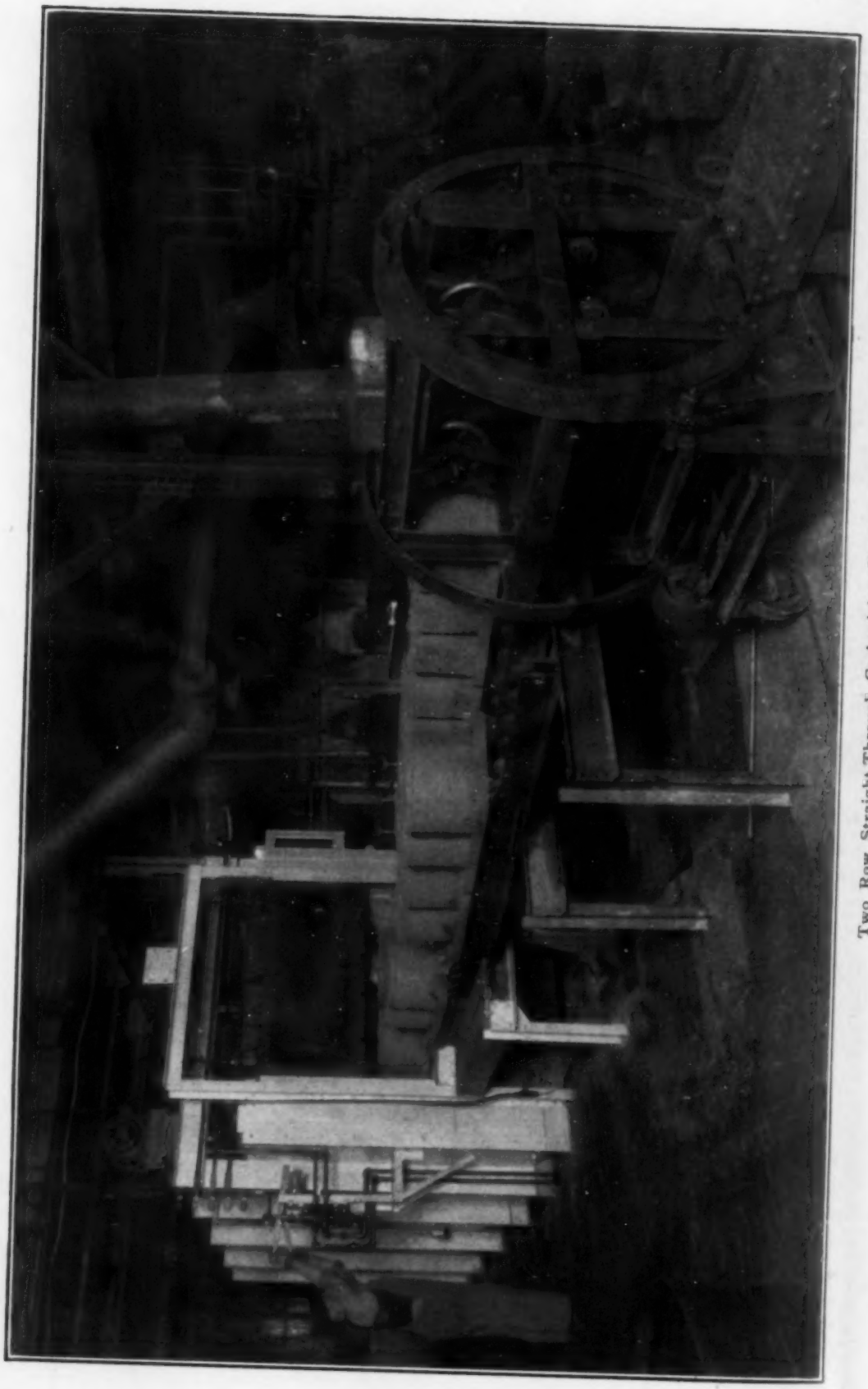
* C. M. Campbell, *Proceedings*, American Society for Testing Materials, March, 1922, p. 495.

horizontal rotating hearth supported on balls on a hardened race and driven by a vertical central shaft. These were large round furnaces 18 to 30 feet in diameter, with hearths 5 to 15 feet wide electrically heated with ribbon units on the side walls. The furnace was under automatic control of temperature and movement of the hearth and usually provided with three temperature control zones in the furnace circuit. The boxes were shoved into the furnace by elevating trucks, previously described, and set on piers. The furnace was divided into sufficient sections around the hearth each representing enough space for one group of boxes. At definite times the hearth would rotate the sector representing one section, and then stop to permit the loading and unloading of the boxes. These furnaces were provided with two doors, one for charging and the other for discharging. An intervening wall was between them so that the hot boxes would not be chilled by those entering the adjoining door. When the boxes were unloaded the hearth then could rotate below the separating wall.

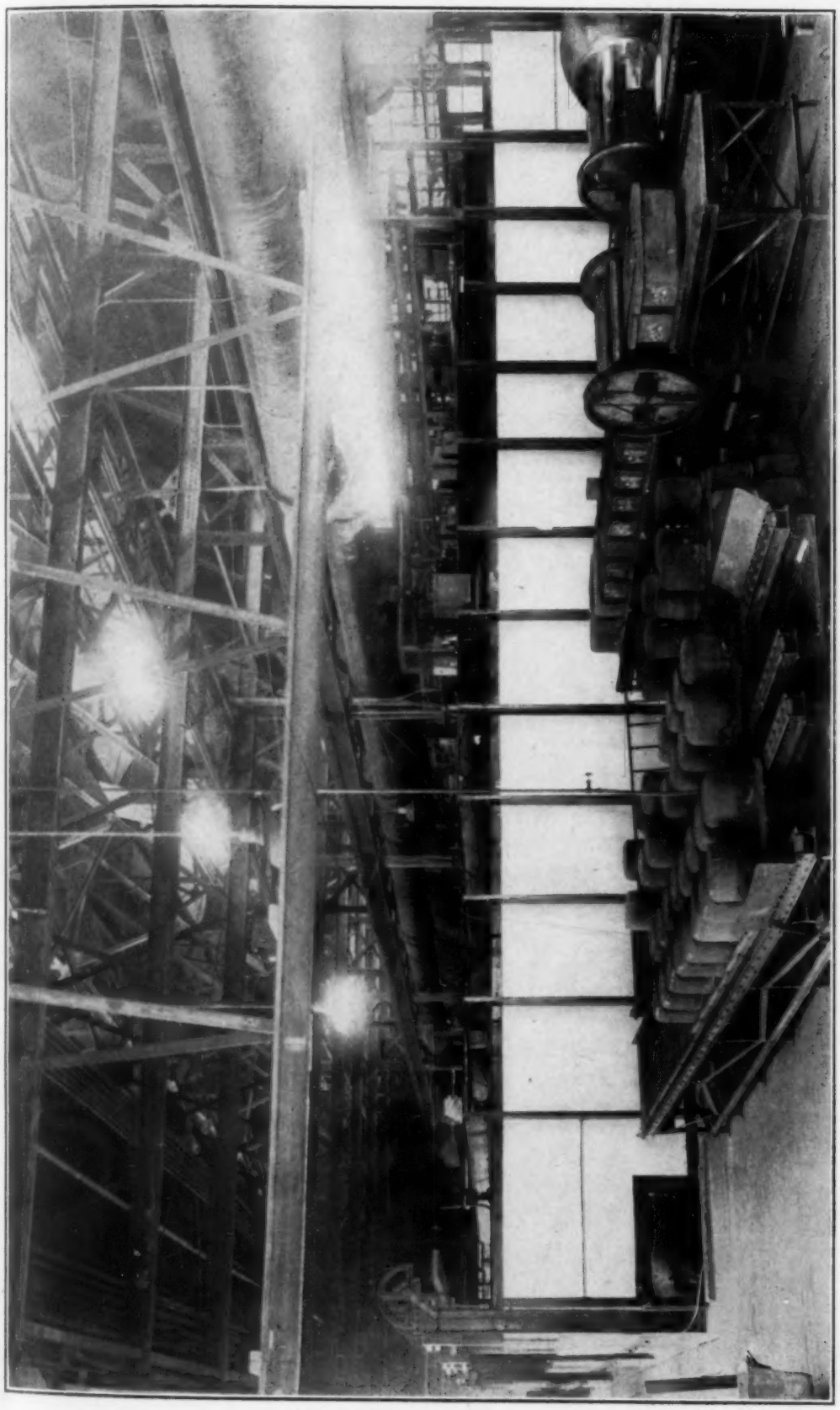
No doubt there was some labor saving due to the concentration of operations and the use of elevating trucks, which in one installation would charge and discharge five boxes at once.* But the boxes had to be unloaded and loaded at intervals. They were not economical in fuel cost because of the expense of electric heating. But there was an immediate improvement in uniformity of product, for each box passed through the same heating cycle automatically controlled and the furnace was at no time chilled by a load of cold boxes. Most of the publicity given these furnaces was relative to their economy, but little was said regarding their possibilities of producing a better grade of carburized work.

After the heat resisting alloy had proven its worth the next furnace design was a long tunnel straight-through type utilizing roller rails cradled on trunnions in alloy shoes which were imbedded into the furnace hearth. The carburizing boxes were laid on trays, set lengthwise or sidewise and pushed by means of exterior mechanical or hydraulic pushers the entire length of the furnace and thus passed through controlled heating zones. These furnaces were of varying width, some having as many as eight rows of rollers so that four rows of boxes could be pushed simultaneously or independently on the various rows. If pushed individually, a separate pushing mechanism was employed for each row. With the latter arrangement a separate cycle could be established for each row and thus

*Dodge Bros.

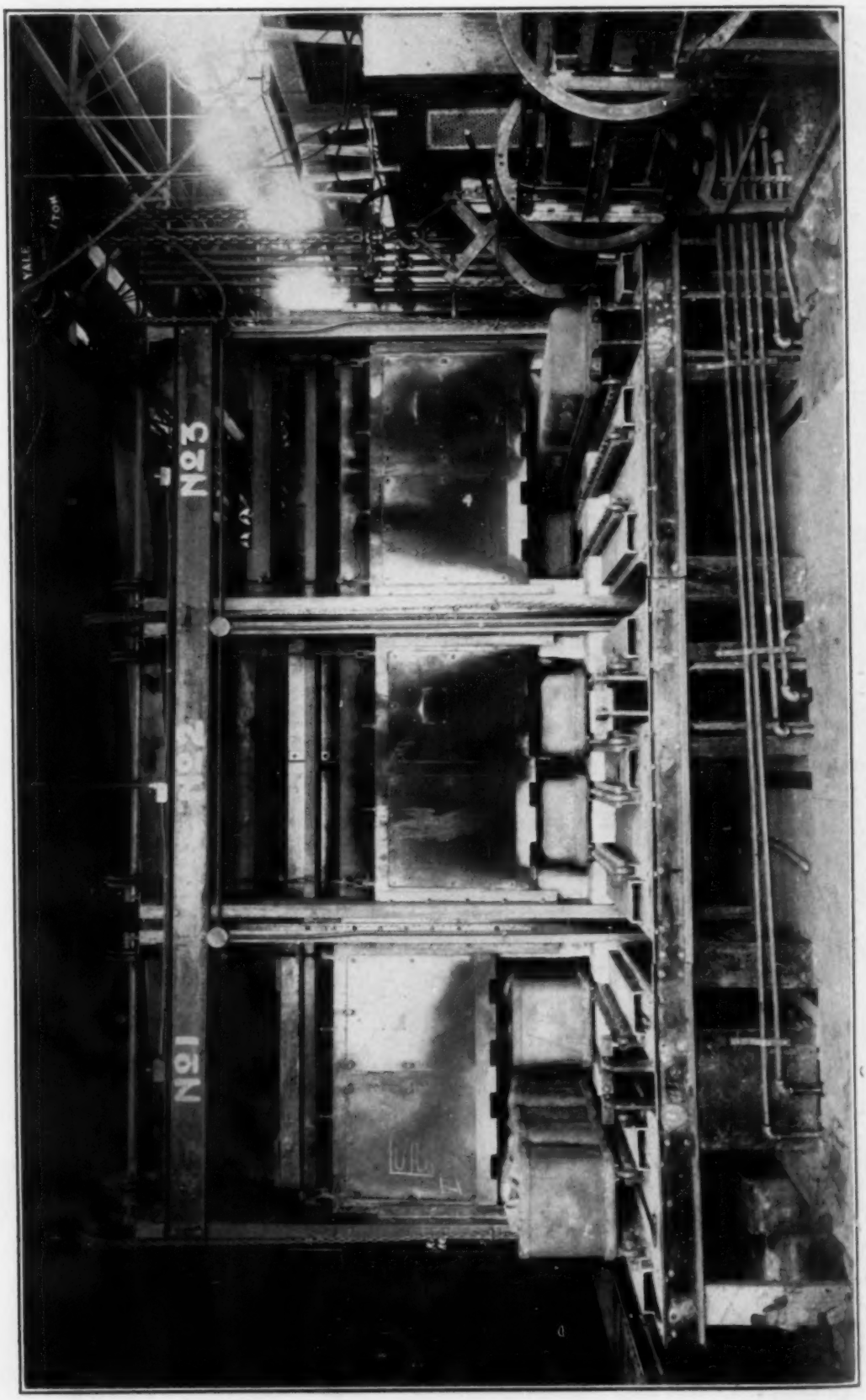


Two Row Straight-Through Carburizing Furnace.

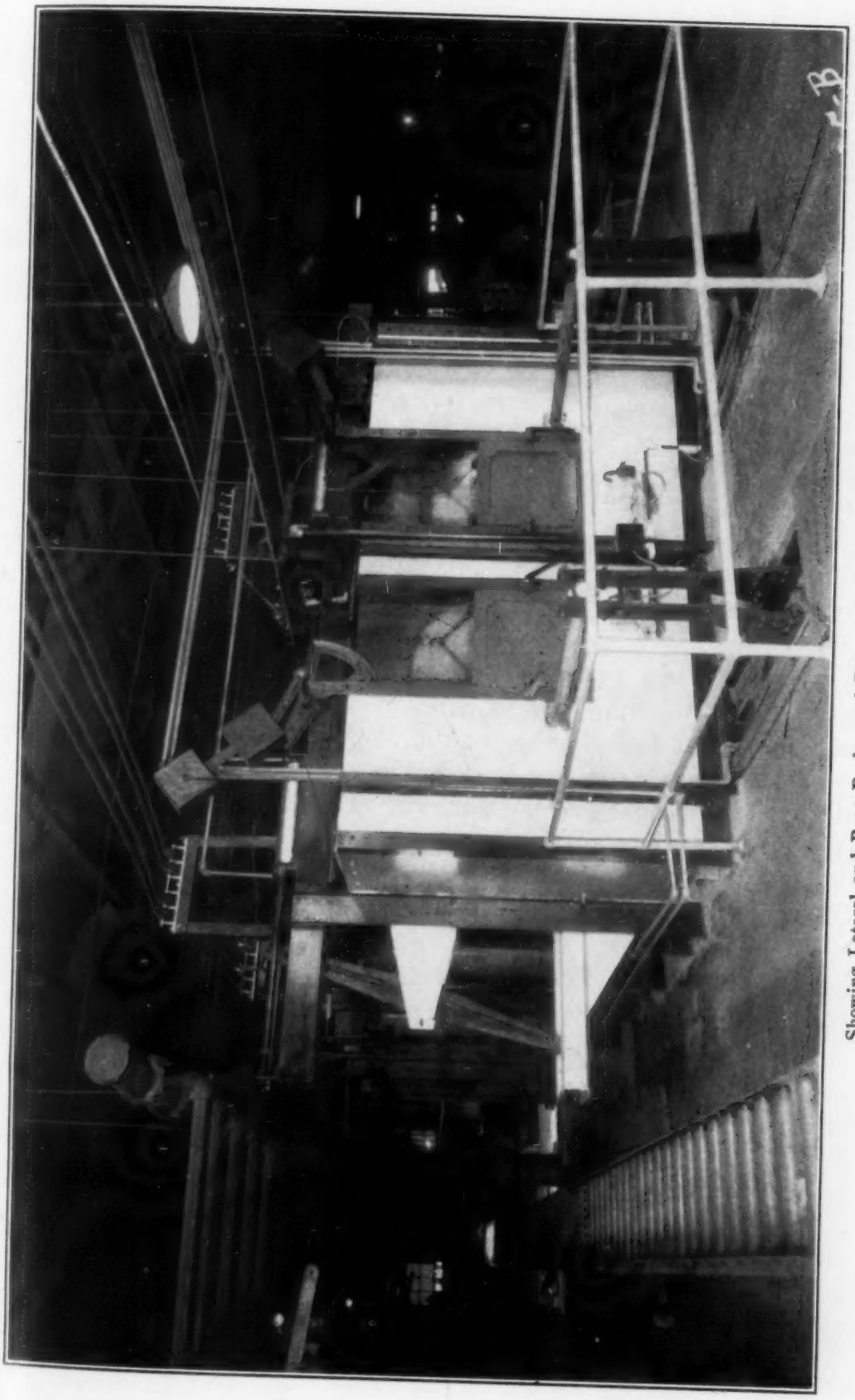


Counterflow Furnace.

Two Row Straight-Through Carburizing Furnace.



Three-Chamber Counterflow Furnace.



Showing Lateral and Rear Pushers of Return Counterflow Furnace.

different depths of case could be obtained. Thus one row might push every 40 minutes when a case depth of 0.060 inch was desired, and another once in 36 minutes when a case of 0.050 inch was requisite. These furnaces were gas, electric and oil-fired, and a large number of them were built from 1923 to 1929.

In one plant* two continuous furnaces replaced 31 oil-fired box furnaces. An estimated annual labor saving of $19,700 was claimed and a total of $45,200 per year over the older method.

Although many metallurgists preferred electric heating, for carburizing for radiant heating is very effective when large masses are to be heated, they were not economical in operation when applied to the tunnel or straight-through furnace and seldom could compete with the gas and oil-fired furnace of this design. The manufacturers of electric furnaces then designed the counterflow furnace whereby the heat ordinarily wasted could be conserved by utilizing it to preheat the boxes entering the furnace. This necessitated that the boxes must be fed in at opposite doors and pass each other, and that a heat exchange chamber must be added to each end of the furnace. The counterflow furnaces were therefore quite long, some 75 feet or more in length. There were two, four, six or eight rows of boxes, each adjoining row moving in opposite directions. This resulted in a long carburizing cycle varying from 25 to 35 hours in the furnace but was quite effective, for the boxes left the furnaces about 700 degrees Fahr. on the outside, and about 900 degrees Fahr. on the inside, thus increasing the fuel efficiency.

In 1928 the first "boomerang" or return counterflow furnaces were installed for carburizing although there had been previous adaptions of this principle for annealing. These are two row chambers utilizing the counterflow idea just described, with the exception that the boxes come out the same door they enter, hence the designation "boomerang." The furnace is divided into two zones. The zone nearest the door which occupies two-thirds of the total interior area is utilized for the heat transfer chamber. The rear zone is the heating chamber, with cast electric units suspended from the arch and below the roller rails.

The boxes are pushed the length of the furnace and at the rear are pushed onto a transfer car connected with a lateral pusher which moves the box to the outgoing rails. The rear pusher then ultimately pushes the box out of the furnace. In actual sequence the rear

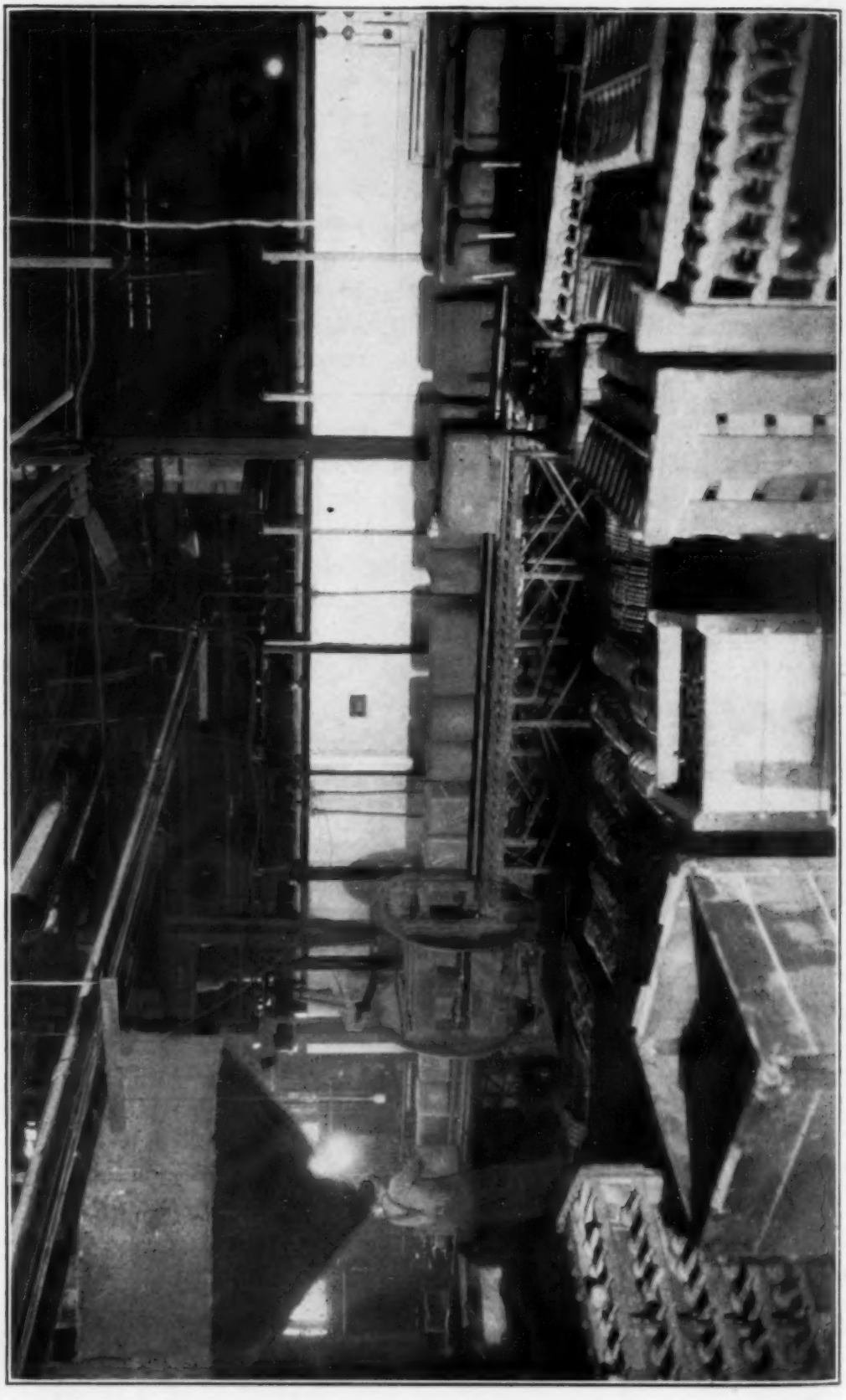
*White-McPherson, TRANSACTIONS, American Society for Steel Treating, Vol. 10, p. 941.

pusher operates first and then retracts. The side pusher moves the transfer car over to the first set of rails. The front pusher then pushes the entire row of boxes forcing the last box on the transfer car. The lateral pusher now withdraws sliding the transfer car on skid rails to the first position. The pushing cycle is now completed.

It would seem this would be too complicated a mechanism to be successful, particularly with so much alloy mechanism in the hot zone. The valve action is controlled by fixed cams in such a manner that the ports directing the hydraulic pressure must open and close in their proper order. Six of these furnaces are in operation in one of these plants, two since 1928. The same alloy is still used in the first installation.

Similar to other pusher furnaces, the case depth is controlled by the pushing interval. Thus a 48-minute push gives 0.040-inch case, a 54-minute push gives 0.050-inch case depth and a 60-minute push 0.060-inch case. All boxes are the same size. This furnace has an advantage in that the boxes change their relative position on the outgoing rails, thus aiding uniformity. These furnaces although essentially the same principle as the standard counterflow have the advantage of less chamber length and occupy less floor space, but have the disadvantage of less production capacity per chamber. The fact that all boxes enter and leave the same door simplifies the handling problem. The cost to carburize in these furnaces is approximately 1 cent per pound but varies from 8 mils to 1.5 cents, depending upon the case depth and type of work.

Parallel with the mechanized furnace has come other improvements which have been labor savers and are equally as important as the furnace itself in economizing this operation. One of these is the roller conveyor for moving boxes to the furnace door. Another is the turn-over machine which enables the box to be reversed, and the third is the mechanical mixing and handling of carburizing compound. The roller conveyor is provided with ball bearings so that the boxes easily slide to the furnace doors, thus taking out all the arduous work in handling heavy boxes. The turn-over machine eliminates the use of clay for luting, the labor of luting and the preparation of the clay. The mechanical mixing enables the quick packing of boxes. The boxes pass on roller conveyors beneath an overhead hopper containing the compound, and by means of a spout the carburizing box is quickly packed. Much fewer men are required now than formerly to attend auxiliary furnace operations.



Showing Carburizing Compound Hopper, Turnover Machine and Roller Conveyors.

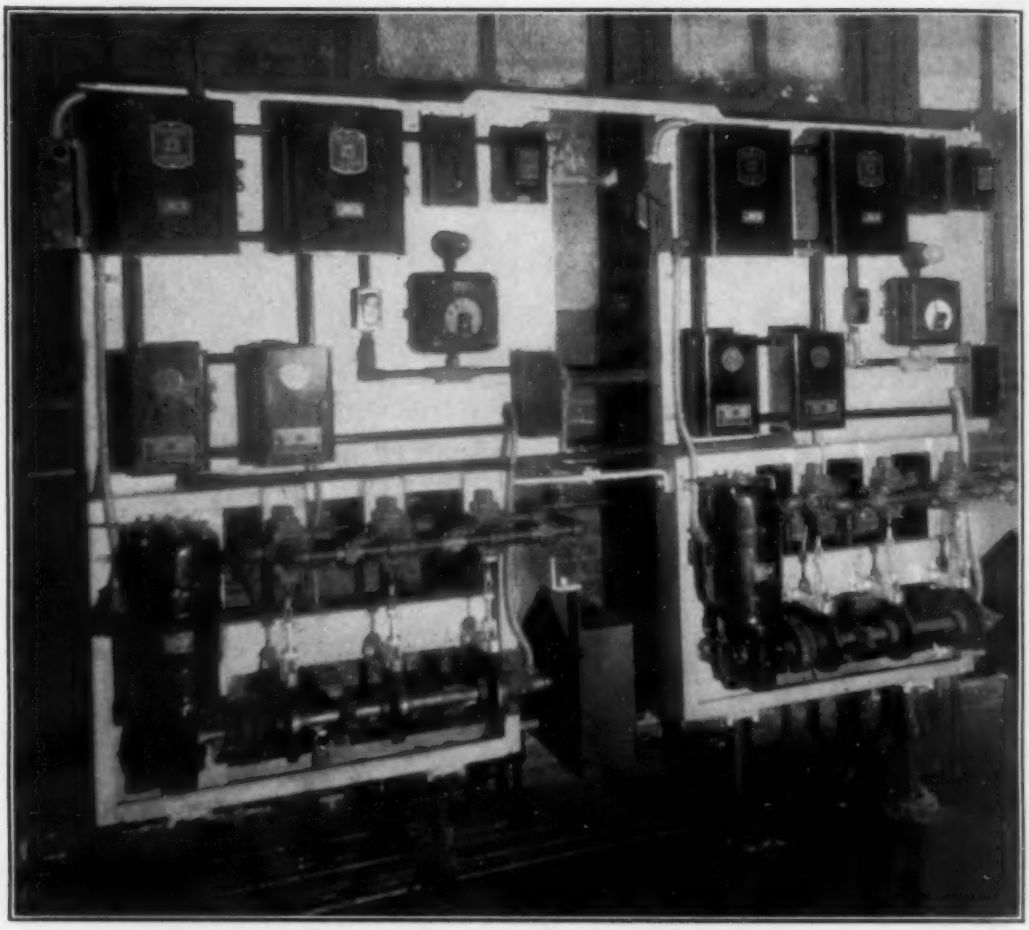
With the introduction of the fine-grained shallow hardening steels it was discovered that such steels would endure prolonged carburizing treatment without coarsening the austenitic grain size and such parts could be quenched directly from the box without further treatment except a tempering operation. Also that such steels did not have a tendency to pile up excessive cementite on the outer layers and furthermore that such steels did not distort any more than the coarser deeper hardening types which had been cooled in the boxes and given a lower temperature reheat. During the last few years the trend of nearly all the carburizing steels has been toward the finer grained steels and a marked increase toward direct quenching procedure. Many of these steels will not coarsen until a temperature of 1950 degrees Fahr. is attained, and the next move may be toward higher carburizing temperatures to shorten the cycle in the furnace. At present many plants equipped with counterflow furnaces have changed them over to straight-through design and are now box quenching.

There is without question some advantage in tooth strength by this method and the simplification of the process has some merit, but the labor economy is somewhat questionable over the cool in box and reheat. It is true that less equipment is required for no hardening furnaces are necessary. However, in compiling the cost of heat treating processes labor is a consequental item. In any hardening operation the only labor involved is putting the piece into the furnace, taking it out and placing it into the quench. To this labor is added the overhead charges which constitute the equipment, supplies, operating expenses, depreciation, etc. For convenience, cost departments determine a percentage based on the labor and which may be from 150 to 1000 per cent. In direct quenching the operations are usually as follows:

Remove box from furnace
Pry off lid
Remove parts from box
Brush parts
Place in quench
Replace lid in box
Remove box to cooling department

To perform this procedure usually two men are required instead of one furnace attendant. It is true we have saved the cost of rehardening, yet bringing the box out of the furnace at 1650 degrees Fahr. and allowing the heat to dissipate is costly also. It is also wasteful

Showing Carburizing Compound Hopper, Turnover Machine and Roller Conveyors.



Cam Arrangement for Controlling Pushing Sequence in Return Counterflow Furnace.

of carburizing compound to expose it to the air during the quenching, and causes its rapid deterioration. To prevent this some firms employ an all-charcoal carburizer which is higher priced. Comparative costs made in one plant where both counterflow and straight-through furnaces were used showed no saving by the direct quench over the box cool and reheat.

It has long been recognized that the reactions occurring between the steel during carburizing has been due to an enriched carbonaceous gas which serves as a carrier of the carbon to the steel. This gas appears to be principally carbon monoxide with carbon dioxide serving as the means of changing carbon to monoxide by the well known reversible reactions. The employment of gas is by no means new. In this country it was introduced in the Machlet furnace about 1910, first built by the American Gas Furnace Co. of Elizabeth N. J. These furnaces consisted of a slowly rotating horizontal retort revolving on rollers and the retort externally heated by gas.

The original principle of operation according to Giolitti* was based on the "Idea of diluting the carburizing vapors of oils at different densities, petroleum, etc., with an inert gas which acts as a vehicle for the carburizing gases, carrying them into the cementation chamber, and preventing their forming a deposit of carbon by their decomposition at the high temperature of cementation."

E. F. Lake† reports that naphtha vapor and ammonia were employed in some of the earlier installations but it is questionable whether it was ever used in commercial hardening.

This method of carburizing did not gain much headway, although there were several installations in operation between 1914 and 1918 which utilized gas under pressure. Although the employment of gas alone was not highly successful in the early days and the application of these furnaces limited to certain classes of work, yet the furnaces were quite effective when the retort was packed with solid carburizer and a large number of them were incorporated in plants manufacturing small parts, such as bolts, screws, nuts, chain links, pins, bearing races, rollers, etc., and many of these furnaces are being used at the present time.

Employed in this manner it was in effect a round carburizing box set horizontally and revolving in a combustion chamber at about 2 to 3 revolutions per minute. These were also provided for means of tilting the furnace so the contents could be discharged into the quench. The chief advantage of this type of furnace was a considerable time over box carburizing methods, and these gave excellent uniformity of case depth.

The principles of gas carburizing as patented by Machlet in 1907 were sound and vary little from those used today, but the furnace was too advanced. The early difficulties experienced when attempting to use gas was no doubt due to the uncertain quality of domestic gas in different localities and probably accounts for some of the early prejudices against this means of carburizing.

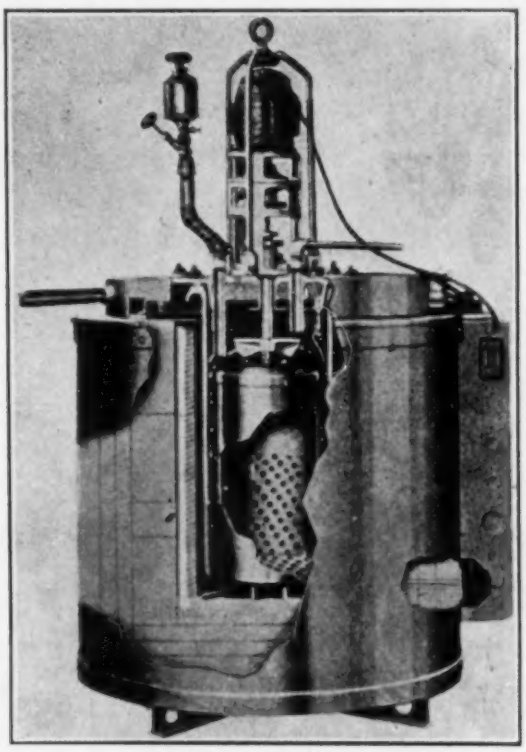
Of recent years there has been quite a few rotary carburizers with horizontal alloy retorts adaptable for small parts and designed to carburize either with gas or solid carburizer. Many of these have a capacity of 500 to 1000 pounds of work per charge, being of larger capacity than the earlier ones mentioned.

The next modification of the gas carburizing principle which

*Giolitti, Cementation of Iron and Steel, p. 290.

†E. F. Lake, Composition and Heat Treatment of Steel, 1911.

attained success was the pit type furnace. These consisted of a round shell, usually sunk below the floor level and equipped on the interior walls with electric heating units composed of round rods recessed in grooves of refractory material. With this equipment was supplied two or more retorts of chromium-nickel heat resisting alloy. The work to be carburized was placed in racks, on fixtures, or in perforated baskets, some holding 800 to 1000 pounds of work and these placed in

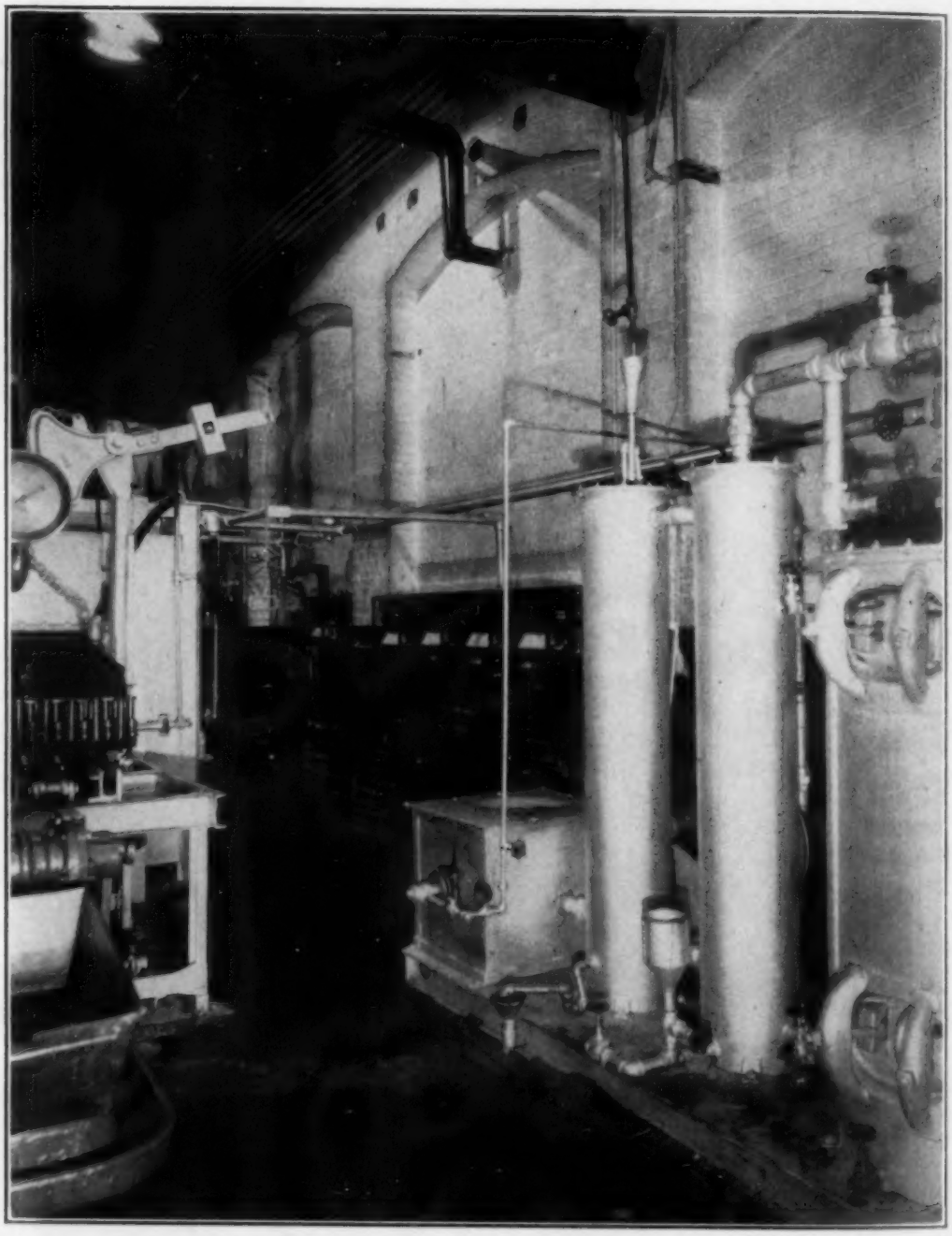


Cross Section of Vertical Retort Carburizer.

the retort. After loading the latter it was lowered into the furnace shell. A sealing cover was bolted on the retort by means of I bolts and fastened to the retort with a collar. On the cover was an electric fan and vertically mounted fan motor and a large drip lubricator. In this cover was also located the thermocouple.

Into the lubricator was placed oil for the carburizing. About one pint of oil per hour of carburizing time was required, or about one gallon per ton of steel carburized. For a charge requiring a case depth of 0.050-inch and containing 800 pounds of work, about two quarts of oil was necessary. Several mineral and vegetable oils alone and mixed have been employed successfully. Some of these are turpentine, linseed oil, bone oil and nitrobenzine with kerosene. In

Battery of Horizontal Rotary Retort Furnaces.



Gas Carburizing Furnace with Gas Conditioning Unit.

some installations propane, city gas and natural gas have been substituted with varying results.

When oil is employed, which is the most common procedure, it is allowed to drip slowly into the retort where it vaporizes. The fan then distributes the vapor throughout the work and the decomposition of the hydrocarbon produces the carburizing gases. For a case

depth of 0.040 inch, five hours are required, for 0.050-inch, six hours and for 0.060-inch, eight hours. These vary some with the size and charge of the equipment but are typical.

When the cycle is completed the retort is lifted out of the furnace shell by hoist and another previously loaded retort is lowered in its place. In the event of direct quenching only the basket or fixtures are taken out.

A large number of firms have installed these furnaces. Some have discontinued these as unsatisfactory; some claim there is quite a difference of case depth between parts coming out of the top and bottom of the retorts. Other users report excellent and uniform results. There is no doubt several advantages to these furnaces, particularly the short carburizing cycle. They have also been used for carburizing very large bearing rings and are adaptable for very deep carburizing which would exhaust the solid compound.

The largest item of cost is the retorts which have an estimated retort life of about 6000 hours and cost approximately $1000.00 each. When a retort cracks and allows air to enter, it generally ruins the entire contents due to scaling. But this bad feature is common to all gas carburizing furnaces using muffles or retorts.

In all of the previous gas carburizers described, the hydrocarbon in the form of gas or vapor was introduced into the furnace without previous treatment except partial dehydration or prior mixing with a certain amount of air. About 1930 R. J. Cowan and associates developed the continuous gas carburizer whereby a hydrocarbon gas is mixed with CO_2 by the medium of flue gas and this introduced into the retort, through which work is progressively moving, with the gases also moving in the same direction. In this muffle are three distinct zones: one for sooting or depositing finely divided carbon on the surface; second, that of active reaction whereby the carbon is converted to carbon monoxide, in a form it is absorbed or combined with the steel; and third, a leaner gas zone whereby no surface activity occurs and which has been termed the zone of diffusion.

A typical furnace for doing this work, including gas conditioning apparatus, cost approximately $15,000. This size equipment consists essentially of a muffle 15 feet long, 26 x 14 inches inside, set into a combustion chamber heated by burners properly spaced for even heating. The muffle rests on cross piers with one end free, and on rollers to allow for expansion of the muffle when heated. The muffle is made of sections of heat resisting alloy. The composition most

favored at present is one of 35 per cent nickel and 15 per cent chromium. This specific alloy has shown the most satisfactory life in service.

In the front portion of the furnace is a charging vestibule and in the rear a holding chamber. The center portion constituting the area of the muffle is the carburizing chamber. There are four doors—two sealing doors on each end of the muffle, and the furnace doors on the extreme ends of the furnace.

The work is moved through the furnace by means of automatically controlled pushers against alloy trays which are pushed on alloy skid rails in the muffle.

The operation of the furnace is as follows: A tray is pushed into the charging chamber with the outer door open and the retort door closed. The outer door is closed and the air purged out of the chamber by the introduction of flue gas. The two muffle doors are now opened and the tray pushed into the muffle. This is continued until the furnace is filled. After a complete charge fills the furnace one set of trays is pushed into the discharge chamber which must be removed before the next push. This is repeated every 30 to 50 minutes, depending upon the depth of case desired. A case of 0.040-inch is possible in four hours and a case of 0.050-inch in five hours total time.

Much of the time which is lost in carburizing by pack hardening methods is consumed in bringing the pots up to temperature. By carburizing directly in natural gas most of this time is saved with a corresponding saving in fuel costs. The labor of packing and unpacking carburizing boxes is eliminated. There is no labor of unloading cars of carburizing compound and using valuable floor space to store it. The cost of the compound itself, which is a considerable item, is saved for the gas cost is negligible. There is no necessity for dust exhaust and carburizing compound mixing and handling systems. There is not the dirt accompanying pack carburizing and these furnaces can be placed directly in production lines. This type of equipment fulfils all the requirements of good carburizing practice and it would seem therefore that this is nearest to the ideal means of doing this metallurgical operation.

However, nearly 40 per cent of the total expense of gas carburizing is in the muffle cost. These cost from $5000.00 to $6000.00 to replace and their life average is placed at about 14,000 heat hours. When these fail the entire furnace is out of commission and must be

practically rebuilt. This usually occurs at a time when the furnace is most badly needed, hence it is often advisable to have stand-by equipment available or the equipment in duplicate so that production will not be held up at a critical time.

Another consideration is the fact that these furnaces always do not carburize as they should and sometimes develop troubles which are baffling to solve. Occasionally there is a tendency to decarburize, sometimes the work comes out covered with soot. At intervals the work will not harden. An austenitic soft skin will occur at times. One firm had trouble with a slight flaking on the surface which was inexplainable. The effect of nascent nitrogen, sulphur and various parasitic gases have not been definitely determined.

These are not given in any sense of condemnation, but many purchase these furnaces and when these troubles occur are inclined to regard gas carburizing as impractical. In fact many of the early difficulties have been solved and more is being learned of the causes. For example, one concern had installed new fixtures for carrying parts through the furnace and found for some reason the parts would no longer carburize. A check up of the gas and equipment showed nothing out of order. After an investigation it was determined that the new fixtures were themselves carburizing and robbing the gas of its effectiveness so that it was too lean to correctly function with the parts. After the fixtures had themselves acquired a case no further trouble occurred.

The cost to carburize by this method is a variable figure, but based on a 14,000 heat hour retort life it would probably be close to the following figures:

Case Depth Inches	Cost per pound—Dollars
0.020	0.007 to 0.008
0.030	0.0075 to 0.0085
0.040	0.008 to 0.009
0.050	0.009 to 0.01
0.060	0.012 to 0.015

As has been stated 40 per cent of the total cost of this operation is in the retort and these figures could easily be doubled if short life of muffles was experienced.

There are other continuous gas carburizers installed in several large plants on much the same general principles but with some modification in design and in the treatment and introduction of the gas, and some differences in the means of conducting the work through

the muffle. In one type, for example, the sooting chamber is not used and pawls or pushing fingers are employed. This design of furnace is also equipped with an elevator quench mechanism on the discharge end of the furnace which lowers the work and fixtures into the quenching bath located below the furnace level. This synchronizes with the pushing mechanism.

Some of the smaller plants, and even larger ones doing a relatively small amount of carburizing, have found it more economical to substitute a stationary box type gas carburizing unit and an auxiliary gas conditioning unit. These of necessity operate a little different than the continuous design.

In these the tray and work are charged into the muffle and the furnace door closed. The air which has entered during the loading of the muffle is displaced by gas made by partly burning 600-B.t.u. city gas with air in the proportion of three air and one gas. The purging operation continues for about 1½ hours as the work is coming up to heat. When the temperature has reached about 1550 degrees Fahr., straight natural gas or its equivalent is now supplied at the rate of about 50 cubic feet per hour. Next the carburizing period is started, during which natural gas diluted with partly burned gas is supplied for three to four hours, or until the completion of the carburizing cycle which would total 4½ to 6 hours, depending upon the depth of case sought. The parts are then withdrawn and quenched.

To determine the relative costs to carburize by any of the present accepted methods is very difficult unless every firm has the same labor rates, the labor efficiency alike, the operations and type of product identical. The published figures of operating costs of some new heat treating development are often misleading. It is frequently the failing of designers of new methods of carburizing to exaggerate their economy, and initial users of such equipment to confirm it, in the ecstasy of early enthusiasm, but later it is discovered their first figures are far from the facts.

Often furnaces which are regarded as economical installations in one plant may be the reverse in another. We do not usually regard carburizing in electric heated box furnaces as the most economical means of doing this operation, yet in one plant they took advantage of the assurance of close automatic control by having the pots loaded by the day shift at the close of the day; set the automatic temperature and time control so that the furnace would shut off automatically at the close of the cycle during the night. In the morning the furnace

was unloaded and another heat charged in the morning. Thus two heats were obtained by one work shift. In another plant two shifts might be employed for the same volume of work.

Some firms install continuous furnaces when their production does not justify this type of equipment, with the result that their furnaces are idle for extended periods. Large continuous furnaces costing from $15,000 to $75,000 can add considerably to carburizing costs if they are not in continual operation, as their name implies, for some of them cost up to $150.00 in fuel expenditure to bring them from the cold to the carburizing temperature.

From a technical standpoint we are primarily interested in uniform and dependable results and the quality of the finished product, rather than whether it can be done for one cent or three cents per pound, hence the cost should be of second consideration. However, those engaged in competitive manufacturing, although agreeing to this, must ponder the economies before an investment is approved.

Within a span of 35 years carburizing costs have been reduced from approximately 15 cents per pound, as practiced in the old coal-fired furnaces, to less than 1 cent per pound, with somewhat doubtful claims of less than ½ cent in some of the newest gas carburizing installations.

Instead of the work being done by crude laborious methods the furnaces and handling equipment have been almost universally mechanized. Where formerly this was one of the dirtiest of the heat treating operations, now modern furnaces are often placed directly in the production lines. Instead of the uncertainty of the human eye in judging temperatures we have delicately balanced instruments controlling not only the temperatures to within a few degrees, but regulating the time cycle as well. Instead of bringing the heat to the box as in box furnace practice, we have the work passing through carefully controlled thermal zones so that each box or individual piece has exactly the same increments of heating. Where formerly containers gave but a few weeks life, we have alloy boxes, retorts, muffles and furnace castings yielding from 1 to 8 years continuous service. Uncertain case depth, irregular carburizing, and a host of defects, which were the bane of carburizing practice for many years, now no longer exist. Some of this may be attributed to the improvement in steels.

In the newest gas carburizing processes not only is the case depth held with exactitude but the hypereutectoid zone can be made as deep as desired, or an eutectoid or hypoeutectoid case produced by con-

trolling the activity of the carburizing gases. This control is impossible by pack hardening methods.

There is no question but that pack carburizing is steadily losing ground by the advent of the retort, pit type and continuous gas furnaces. The increasing number of gas carburizing installations, that have gone into the larger as well as the smaller plants, is evidence of the inroads this method is making. Pack hardening is an antique heirloom from the past, and the process of carburizing has progressed in one generation more than it did in all the previous centuries. However, we have always carburized with gas, for the luted box filled with carbonaceous material is essentially a small individual gas unit capable of slowly generating sufficient active gas to accomplish the phenomena of carburizing.

DISCUSSION

Written Discussion: By H. W. McQuaid, metallurgist, Republic Steel Corporation, Cleveland.

This presentation by Mr. Davis is enjoyable to one who, like myself, has been actively interested in commercial case hardening for more than twenty years. These twenty years have seen the most important transition in carburizing practice, although if we exclude the part played by heat resisting alloys the fundamental changes have been very small.

Today, in the United States, it is possible to find in operation practically every type of carburizing described by Mr. Davis, from that type which was common back in 1800 to those of all the intervening types. There are carburizing plants today operating in the United States using raw bone and carburizing at temperatures from 1500 degrees Fahr. upward. There are not in this country, to my knowledge, any furnaces which are fired by coal, but these are still used to some extent in Europe. Many carburizing furnaces fired by oil without the aid of a pyrometer are used for carburizing in which various compounds are in use with cast iron pots. In fact, some rather large installations still use cast iron or cast steel carburizing boxes because of the intermittent nature of their requirements.

Many of the carburizing plants today, especially those on a smaller scale, are exceedingly dirty, poorly lighted, cold in the winter time and almost unbearably hot in the summer time. Invariably this type of equipment is supposed to be in the condition it is in order to avoid high production costs and probably if the truth were known many such plants could be more economically operated by sending their carburizing work to the nearest commercial heat treater.

Where the production volume warrants it, much thought can profitably be given to the design and layout of the small case hardening department consisting of one or more carburizing furnaces. The location of the carburiz-

ing furnaces in reference to the hardening furnace and quenching tanks should be given careful study.

The layout of quenching tanks which will permit quenching not only from the hardening furnace itself, but also from the carburizing boxes, is of primary importance. The improved quality of direct quenched parts, which has been made possible by the development of fine-grained steel, has made the direct quenching of carburized parts of primary importance as indicated by Mr. Davis. The advantages of direct quenching fine-grained steel has been overlooked by many of the operators of smaller carburizing departments. This is explained in some cases by the difficulty in obtaining the fine-grained type of steel where the amounts purchased are small, but where it is possible to insure steel of the correct type, quenching direct from the box can be substituted in most cases for the more expensive reheating operation. Proper protection for the hot carburizing boxes, to reduce chilling effects and to protect the operator while he is removing the work, is usually simple to design and should not be omitted. An investment of a few dollars in a sheet asbestos protective screen will be one of the best investments which could be made in equipping a carburizing department.

Consideration should be given to the use of floor level furnaces as compared to the elevated hearth type. The floor type furnace is especially suited to the handling of large carburizing boxes by means of hand trucks and permits easy removal to the quenching tank. The installation of conveying equipment both for handling compounds and boxes warrants considerable attention and when properly designed is usually of great advantage.

We should not be too hasty in our selection of a gas carburizing furnace to replace the more common standard oil-fired furnace. It is quite common to compare costs on a very modern, well designed gas carburizing furnace with an extremely obsolete, poorly designed fuel-fired type of furnace. It is my opinion that, for the usual run of material a modern, properly designed oil-fired carburizing furnace, using carburizing boxes which are designed to reduce the insulating effect of the compound to a minimum, will produce carburized work about as economically as the modern gas carburizing furnace. Oil-fired furnaces can be designed to give uniform temperature distribution and will supply the necessary B.t.u.'s more cheaply than any other form of fuel where fuel oil is accessible. There are, of course, some exceptions to this statement, but in my opinion we should not be too hasty in eliminating the use of fuel oil as a possibility. With a modern oil burner and fuel handling system and a well designed and insulated furnace using the correct materials for hearth brick and piers, results can be obtained which will be satisfactory for practically any commercial carburizing operation. There are special cases where gas is relatively cheap and in these cases the advantages of gas in obtaining uniform results with less effort would warrant the selection of gas as a fuel.

One of the most important factors in determining the results which can be obtained with various types of equipment is that of design of the carburizing container. As pointed out by Messrs. Roush and Dames, this is an important factor in reducing the overall time of carburizing and it has been my experi-

ence that by the use of sheet alloy boxes, designed to conform to the work as described by Messrs. Roush and Dames that it is possible in oil-fired furnaces to deliver carburized work of approximately 0.050 inch case in as short a time as it can be done with the usual gas carburizing furnace.

The gas carburizing furnaces are particularly well adapted for the production of light cases as developed for one of the large automobile producers. They are especially well adapted for applications where control of the carbon in the case is of importance, although this is difficult to obtain in the continuous type of furnace.

The batch type of gas carburizing furnace, particularly the rotary type, lends itself to the production of carburized cases with a maximum carbon content controlled to meet most any specified amount. This can be done by a proper balance of carburizing and diffusing time. The intermittent flow method of alternate carburizing and diffusing periods is probably the most satisfactory since it permits more accurate control of the carbon content and eliminates the soot problem.

Before making an investment in an expensive gas carburizing layout, it would be well to consider what the most modern compound type of carburizing equipment has to offer. Before selecting a given type of gas carburizing equipment, careful consideration should be given to the restrictions imposed by the design of the product to be carburized so that uniform carburizing can be assured wherever required. Some types of gas carburizing furnaces give difficulty due to the nonuniform cases because of lack of uniform gas conditions, which result from insufficient circulation. Where parts contain deep recesses requiring good circulation of the carburizing gas, consideration should be given to the pit type of furnace which employs a circulating fan. As much care should be given to the design and layout of a carburizing department as would be the case with any other department of equal importance. All furnace and other equipment should be bought to produce the particular product involved, and care must be exercised in order to prevent a carburizing department being built around a furnace rather than around a given product.

Written Discussion: By John F. Wyzalek, chief metallurgist, Hyatt Bearings Division, General Motors Corporation, Harrison, N. J.

The author's review of past practices brings back recollections of days when the writer, during period of 1913-1916, had occasion to use considerable quantities of carburizer, consisting of the very complicated ingredients of raw bone, bone black, charred leather and charcoal. It proved to be a very expensive one as compared with a coke-charcoal energized compound later adopted.

Although it is true that during recent years the trend has been toward direct quenching, a word of caution is in order to guard against use of such procedure wherever the parts involved require minimum retained austenite for satisfactory performance. Also, although such parts as gears may offer less distortion by direct quenching, on the other hand, small parts like, for example, cylinders, it might prove more economical to double treat with quenching fixtures so as to better control size and shape, thereby effecting a saving in grinding costs.

In connection with gas carburizing developments, it is felt that to the

imposing list should be added the recent development of the so-called "Nicarb" process by Machlet of American Gas Furnace Company. Through the use of an atmosphere of a carburizing gas, such for instance as propane, combined in proper proportions with ammonia and air, it is possible to produce depths of case up to at least 0.015 inch, in character similar to a cyanided case, file hard, using an S.A.E. 1010 steel without quenching and with minimum distortion.

Written Discussion: By Gordon T. Williams, metallurgist, Cleveland Tractor Co., Cleveland.

Mr. Davis' fine paper shows the broad comprehension of the subject that we rightfully expect from him. The author emphasizes the importance of muffle cost in gas carburizing; this is the largest variable, and, with all credit to the suppliers of both furnaces and alloys, the greatest handicap in the use of many types of equipment. Cylindrical retorts give high average life and good dependability, probably because of their simple shape and the experience which has been obtained in their casting. The various horizontal mufflles still offer many problems, particularly in the elimination of crevices where carbon may accumulate and by wedging cause cracks. We have had muffles fail by some such mechanism after only a few hours' service. However, we frequently find that the cracks can, by manipulation of the cycle, be "sooted" so that carburizing can be carried out for as much as 5000 hours after first cracking. Other muffles will, after a short or long life, open up so much in a period of a few hours that they can no longer be used. Elimination of flanges and joints, as well as improved furnace design and foundry practice, should raise the expectancy of service. In batch-type horizontal gas carburizers, the localization of thermal stress due to effect of a heavy cold charge on the muffle bottom is severe.

Another phase of the alloy question to be considered is that gas carburizing in stationary furnaces may demand more rather than less alloy equipment (and cost) than box carburizing, due to the supports, fixtures, and trays required, and these will tend to be of low durability.

We carburize, in a normal month, over 250 tons of material by gas. Batch-type units were selected for this work because of their far greater flexibility for parts of widely varying weight and case depth requirements; continuous furnaces demand a reasonable regularity of work as regards quantity, section, and case depth.

Written Discussion: By J. A. Dow, engineer, Holcroft & Co., Detroit.

We have read this paper by Mr. Davis with a great deal of interest, and are happy for the opportunity to express an appreciation everyone must feel for the fund of information he has collected and presented.

His history of the development of carburizing is very timely, giving a background to this period of rapidly changing methods.

We would like to add one observation to his treatment of operating costs, particularly referring to carburizing with gas in continuous furnaces. The hourly operating cost of any given gas carburizing furnace is almost constant irrespective of whether the furnace is operating at full or half its rated capacity.

For this reason the furnace size should be selected with a great deal of care when new equipment is being installed.

Written Discussion: By O. W. McMullan, metallurgist, Youngstown Sheet and Tube Co., Indiana Harbor Works, East Chicago, Ind.

The author has compiled an interesting history of the development of the carburizing process. The records of men's success after many failures and the evolution of scientific knowledge from ignorant superstition inspires us to new efforts but at the same time reminds us that there may still be room for improvement and new developments will be forthcoming. The progress in the present generation is probably largely due to the fact that the cloak of secrecy has been removed and information has been exchanged freely. The metallurgical profession may well be proud of being a leader in this exchange of information and the rapid advancement that has been made.

Furnace equipment, methods of handling, cost, etc., have been quite fully discussed in the paper but less mention is made of the quality of the work from different processes under normal operation. Gas carburizing has reduced total carburizing time. Perhaps this is mostly due to shorter heating up time and the work may be at the carburizing temperature approximately the same length of time. With the use of different media, however, it might be expected some difference in time would result and differences in character of the case also as it is known that some media will produce higher surface carbon contents than others. I would like to inquire if the author has associated any noticeable differences in physical properties of the case hardened parts resulting from such things as carbon concentration or steeper carbon gradients arising from the various methods. Lack of oxygen absorption and greater normality of structure has been reported when carburizing in certain gases instead of solid compounds. Are such gases under a disadvantage when using steels of the more or less abnormal type whose properties are considerably dependent on the carbide formation?

Author's Closure

Mr. McQuaid's remarks in reference to the advantages of oil-fired furnaces is worthy of consideration. Manufacturers of alloy boxes almost always agree, however, that the life of alloy is much reduced when furnaces are oil-fired. Perhaps as Mr. McQuaid implies, if sufficient thought was given to the design of oil-fired furnaces a considerable improvement in combustion could be effected. The oil-fired furnaces of most of the early designs were quite expensive in maintenance and little attempt was made to place the fuel under automatic control. We have found that furnace life was improved in either gas or oil furnaces when automatic control was applied. Also the consumption of fuel was materially reduced.

Mr. Wyzalek has brought up some very interesting comments regarding retained austenite and we agree that there is no simple process for heat treatment which will have universal application. The same is true of direct quenching from carburizing. In many instances it might be advisable to cool slowly and reharden or to utilize a second treatment after the direct quench.

Mr. McQuaid has said in discussion of his paper, "It should not be necessary to make the metallurgist subservient to the furnace." In other words the furnace should be able to give us what we want and should be flexible enough to produce the type of product which we desire and not have to adjust our

metallurgy to the equipment. Any equipment is not satisfactory unless it will do the specific job successfully, and we should not let our enthusiasm for novel processes lead us into the wrong channels.

Mr. Williams has advanced an interesting thought on the design of muffles. The cylindrical muffle, no doubt, has a greater expectancy of life than the square or rectangular type, and yet it seems the retorts on nearly all the continuous types are rectangular and have numerous joints and corrugations in them. Yet we know from our experience that cylindrical carburizing boxes used in pack hardening will outlast the square and rectangular design.

Much of the trouble we have had with retorts has been due to the joints separating on account of irregular heating and cooling, and this same local differential in heating and cooling has often been responsible for cracking and short muffle life. Muffles with corrugations are sometimes quite difficult to clean of the accumulated carbon. No doubt, if the manufacturers of these furnaces would give more consideration to a round retort or muffle, these might have longer expectancy, and muffle life is important in carburizing cost by the gas method.

Another item of cost in gas carburizing not included in my paper is the multitude of small fixtures which are necessary to handle the various kinds of carburized parts. This confirms Mr. Williams' statement that you would use far more alloy in gas carburizing than by pack carburizing.

Mr. J. A. Dow has expressed a warning that the furnace costs will be constant whether a gas carburizing furnace is completely filled or only partially filled, hence the production variables should be carefully studied when a new installation is contemplated.

In reply to Mr. O. W. McMullan's question regarding any effect of steeper gradients when the work is carburized in different media, we find it depends upon the specific application of the part. Parts which are subjected to impact, such as clashing gears, will show a tendency to spall if the carbon content is too high on the outer layers. There is also a greater tendency for surface cracking during grinding when the carbon concentration is too high. On the other hand where the element of wear is paramount, a high carbon case does not appear to be detrimental. It is also realized that temperature of carburizing and the type of steel used are important factors in the amount of carbide concentration which occurs. I believe it is generally recognized that a well diffused case is preferred for most purposes. One firm endeavored to introduce a process which would produce 0.040 inch case in 1½ hours. They were able to do this, but uniformity was lacking in case depth and extremely high concentration was noticed on the outer layers.

Mr. McMullan inquires whether steels of the abnormal type might cause some difficulties when carburized by means of gas. There is some difference of opinion on this. One metallurgist claims that when carburizing steels of the Amola type in gas, using parts such as gears, that the gases do not penetrate successfully into the recesses, such as the bottom of the teeth, and show considerably less case depth at the root of the tooth than on the tip of the tooth. However, nearly all the standard fine-grained alloy steels are carburized in gas and most of the results reported do not indicate much difference in the results in comparison to box furnace practice.

A STUDY OF COMMERCIAL CARBURIZING CONTAINERS

By R. W. Roush and A. C. Dames

Abstract

This paper describes briefly the evolution of the carburizing container from the original heavy cast iron type to the modern individual unit type using thin sections of heat resisting alloys designed to reduce the insulating effect of the compound to a minimum. This results in an important reduction of the time of heating and insures improved uniformity of temperature throughout the work. The various types of alloys used for carburizing containers are indicated and the factors which influence their service life are discussed. The importance of scientific container design is stressed, as well as the importance of factors commonly overlooked, such as furnace design, refractories, and fuels.

Examples are shown from practice, indicating what can be done in designing containers which conform closely to the work in compound carburizing. There is a brief discussion of retorts for gas carburizing furnaces as well as containers for liquid carburizing.

Introduction

ECONOMIC uncertainty, with the continued rise in production cost, has increased the demand for new and improved methods that will provide greater efficiency in the heat treatment of steel parts. Because of the highly specialized nature of heat treating equipment, with its attendant high cost, the question of replacement must be decided on the basis of whether or not the new equipment incorporates features which will result in a maximum saving with a minimum expenditure.

During recent years, through the combined effort of engineers, metallurgists, and heat treaters, the development of many heat treating operations has shown continued improvement in quality, as well as a reduction in cost; while carburizing, one of the oldest of the arts, has shown relatively little change during the present century.

A paper presented as part of the Symposium on Carburizing presented at the Nineteenth Annual Convention of the American Society for Metals held in Atlantic City, October 18 to 22, 1937. Of the authors, R. W. Roush is chief metallurgist, and A. C. Dames is superintendent of heat treatment, Timken-Detroit Axle Co., Detroit.

The progress made through the development of continuous gas carburizing, recently introduced commercially, has resulted in a number of special installations which have replaced other methods of carburizing in a few of the larger plants, but the greater portion of parts requiring this type of treatment is carburized in containers with solid compounds. Furnaces of modern design, particularly those of the continuous type, have improved the carburizing practice; compounds have been refined, but the container equipment in most plants is still of the conventional type, either cylindrical or rectangular in shape and as large as can be conveniently handled.

Since the introduction of heat resisting alloys, few fundamental changes have been made in container design, but the results of gradual improvements have demonstrated their importance, and today there is a growing appreciation on the part of metallurgists and production heat treaters that the proper work-container relation is of primary importance to the success of the process. Unlike most heat treating operations where convection as well as radiation heating is possible, parts to be carburized by any of the commercial methods in use can be heated only by radiation and conduction, with an accompanying increase in fuel consumption.

Experience gained from tests taken with a number of different types of carburizing pots and boxes affords definite proof that where the important factors such as proper container-furnace relation and work-container relation are embodied in box design, the cost of carburizing can be substantially reduced without impairment of quality.

History

The earliest carburizing has been treated in interesting accounts by a number of writers. Descriptions of carburizing processes, with a surprisingly great variety of compounds used, are more numerous than accounts of containers.

The modern machine, particularly the automobile, brought about the development of the earliest needs for quantity carburizing. Previous to the sale of machinery to the public, i.e., the automobile, the sewing machine, the typewriter, etc., the amount of carburizing done was negligible, and the cost relatively unimportant. The time element did not matter and the amount of work done was so small that containers were not a serious problem.

The first carburizing containers were largely direct copies of the

cast iron or cast steel boxes previously used for annealing. They were characterized by massive sections, up to two inches thick, sharp corners, and ill-conceived arrangements of both legs and covers. Covers were usually fitted into the open top and plastered with clay or "luted." Whenever carburizing containers were used in reasonable quantities, such as in the sewing machine, ball bearing, and textile machinery industries, various experimentation was done with mixtures of iron to create resistance to oxidation and longer life. Silicon iron in various percentages was employed, which in certain small and heavily constructed containers withstood cracking long enough to render a considerable improvement over ordinary iron.

Heavy cast iron containers, boiler plate boxes, oil well casings, and secondhand pipe, were doing most of the carburizing in 1914, and were being partly replaced by steel castings which showed a slight advantage through reduced cracking.

The first commercial heat resisting alloy carburizing containers sold in America were a by-product of heat element wire manufacture and the remelting of wire scrap. The composition, 80 per cent nickel, 20 per cent chromium, which was the highest grade heating element wire, was inclined to be soft and ductile and lack stiffness and sustained strength at carburizing temperatures. Variations in this composition were tried, which led to the development of the heat resisting alloys used for our modern containers.

Most of these early alloy boxes were used under conditions not at all conducive to good performance, the old style furnace being characterized by coal firing, tremendously uneven heat distribution, flame impingement, uneven hearth bottoms, and numerous other factors which exerted very severe influences of an adverse character on the containers. It is remarkable that such units employed under these conditions demonstrated the economy and the relatively satisfactory performance which they did.

Better performance of the nickel-chromium alloy containers brought about a reduction in weight and wall thickness. Cast alloy containers are now ⅜ to ½ inch thick in sections which were 1 inch thick and more in the early containers.

Figs. 1 and 2 show some of the earlier types of cast heat resisting alloy containers. Because of the excessively heavy metal sections, shrinkage voids were frequent, and further because of the sharp corners, stresses were concentrated at these corners, and the normal performance of such boxes, while definitely superior to cast iron, was

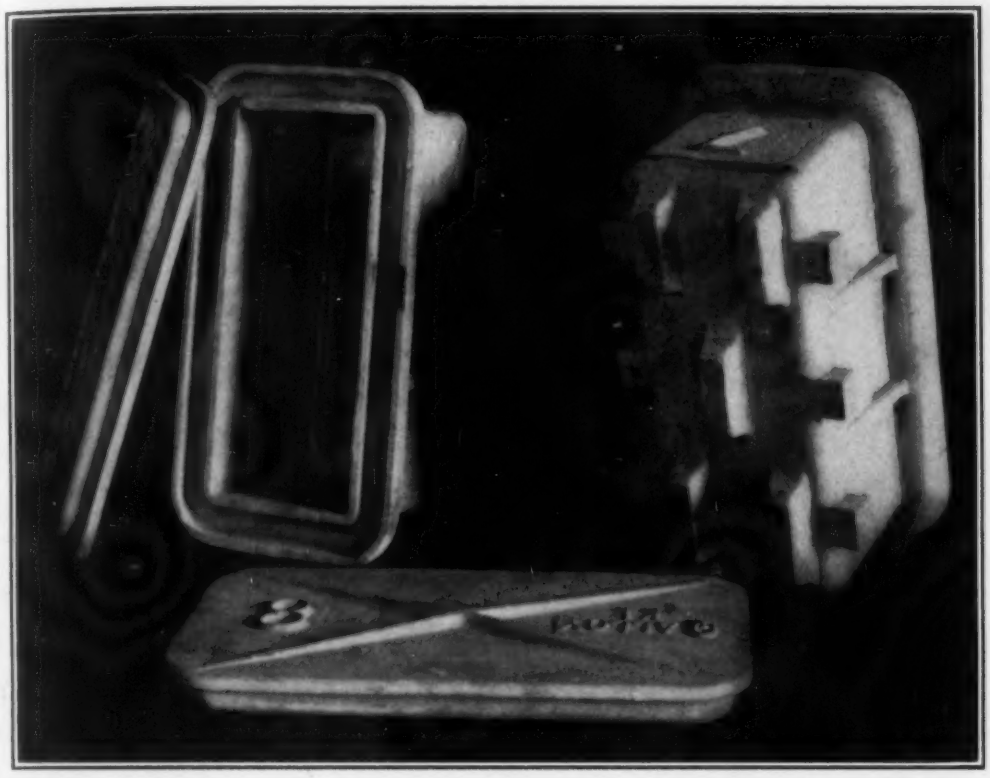


Fig. 1—Photograph of One of the Earlier Types of Cast Heat Resisting Alloy Containers.

not as good as it should have been and did not reflect the full possibilities of the alloy. Their performance nevertheless was so superior that there was no question about their definite applicability to the service and to their eventual and certain economy. The heavy wall section, sharp corners and the leg arrangement of the inverted type box are shown in Fig. 2.

Fig. 3 is interesting, in that it shows a type of cover design intended to provide tight sealing through the beveled interface between the pot lip. Expansion of the pot and settling of the cover on reaching carburizing temperature proved disastrous, in that when subsequent cooling occurred, the cover was wedged in the pot, straining the lip, with the resulting eventual breaking away of the lip from the pot.

Fig. 4 is an illustration of an early type chimney pot for carburizing ring gears. This was an improved design with the chimney cast integral with the pot. The heavy wall section is still in evidence.

About the year 1926 and 1927, the sheet container became quite popular, and has met increasing use by some heat treaters, while others have returned to castings, due to special requirements. Every

Fig. 2—Photograph of One of the Earlier Types of Heavy Wall Section Inverted Type Carburizing Container.

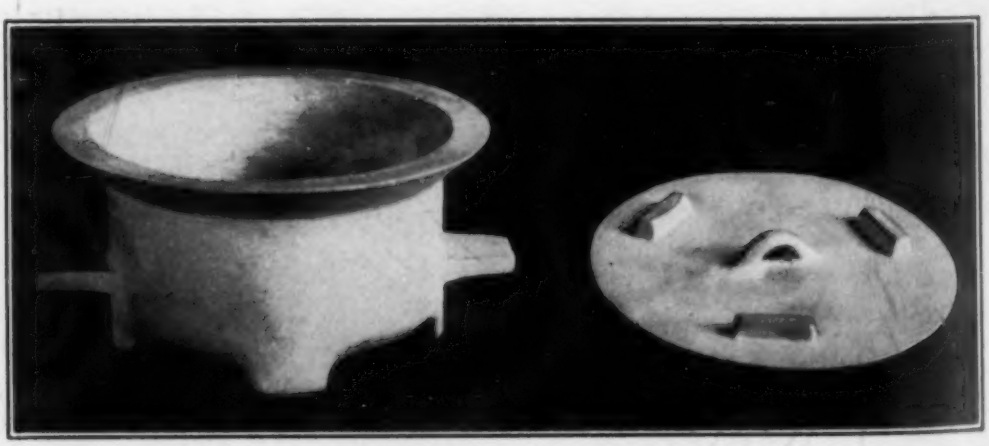


Fig. 3—Cast Heat Resisting Alloy Carburizing Container Having a Cover Design Intended to Provide Tight Sealing Through the Beveled Interface Between the Top Lip.

Fig. 4—Photograph of One of the Earlier Types of Carburizing Container for Ring Gears Having an Inner Chimney.

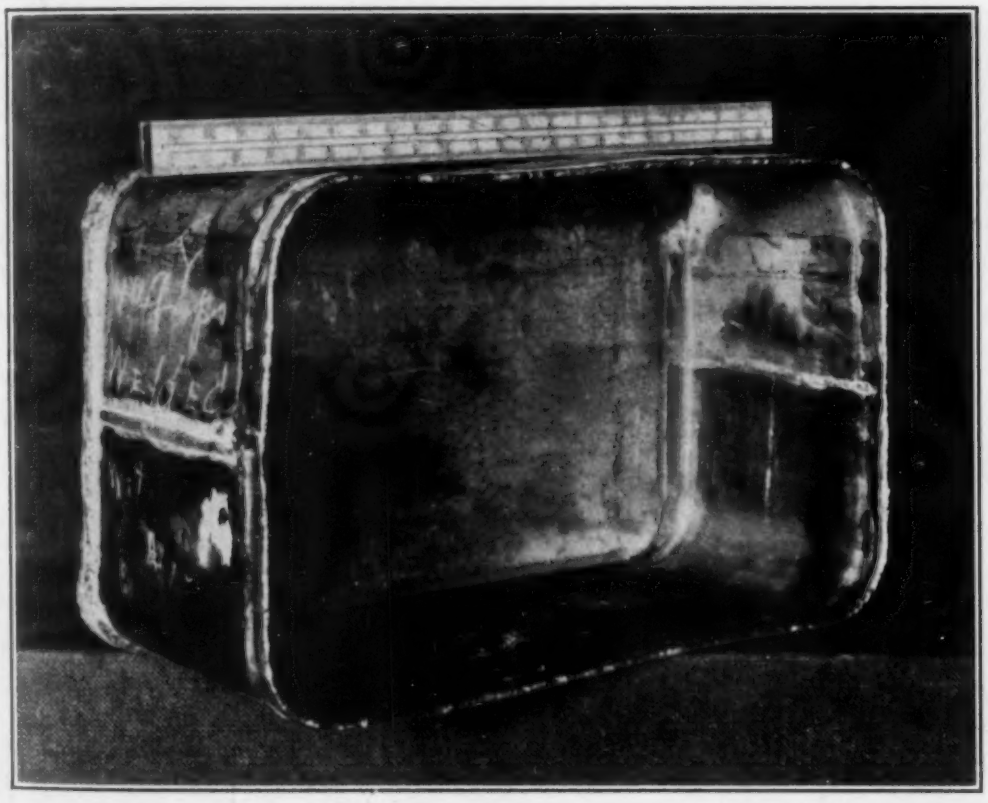


Fig. 5—Photograph of an Early Type of Sheet Carburizing Container.

conceivable arrangement of fabricated and welded sheet containers and combination sheet and cast containers has been tried. Today the sheet container is used extensively in certain applications, and has shown equal economy to that of castings. Fig. 5 shows an early

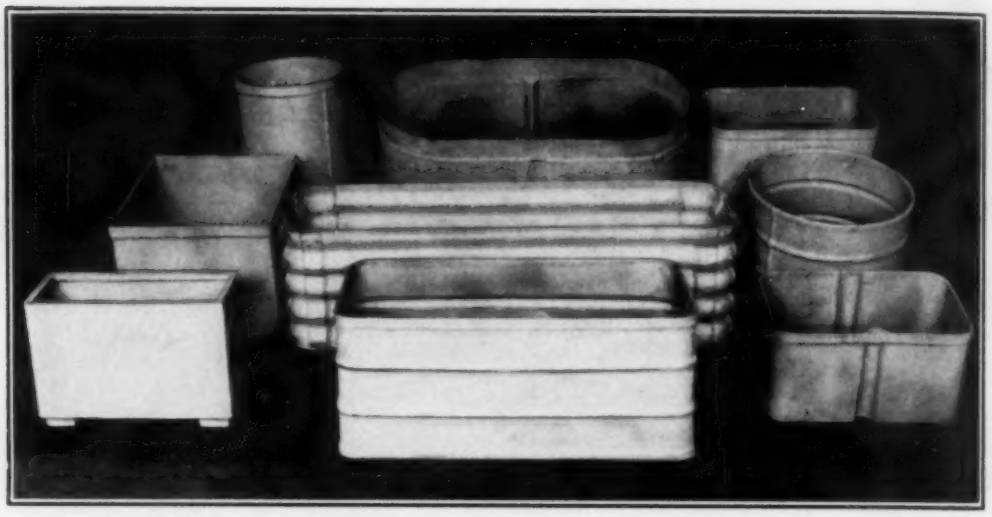


Fig. 6—Photograph Showing Several Types of Combination Carburizing Boxes in Which the Sheet Sides were Cast Into the Corners.

type of sheet box. The sides were formed in two parts and welded together at the ends, and the bottom welded on.

Fig. 6 shows a type of combination box in which the sheet sides were cast into the corners. The base and cover were cast and fit loosely on the box. This type of box gave good performance in certain applications, and is in use today.

Modern Heat Resisting Alloy
Composition, Structure, Physical Properties

The chemical composition of some of the most widely used heat resisting alloys is given in Table I. To say that any one is best for

Table I

No.	Per Cent			
	Carbon	Nickel	Chromium	Silicon
1	0.17	8.80	19.0	1.95
2	0.20	10.90	24.3	....
3	0.15	10.90	24.8	2.26
4	0.11	26.25	18.2	2.54
5	0.10	20.40	25.4	1.52
6	0.23	.....	20.7	....
7	0.20	35.70	15.8	....
8	0.28	40.25	21.7	....
9	0.42	60.70	13.2	....
10	0.60	65.00	16.5	....

all applications is a matter of opinion. As a general rule the higher the percentages of nickel and chromium, to certain limits, the better the heat resisting properties. While this statement is true, a com-

parison of the costs of these alloys shows that the alloy No. 10 in the above table, must give 1½ times the service life of the lower priced alloy No. 7, in order to show equal economy.

It has been stated that nickel in excess of 20 per cent greatly retards the absorption of carbon. While this is true to a certain extent, containers having as much as 65 per cent nickel have been found to be carburized to a depth of ¼ inch after extended service. The nickel and iron contribute strength and toughness to the alloy. Chromium and silicon help to form the hard adhering oxide coating and contribute to the chemical stability of the alloy. Chromium and iron alloys are not stable and carburize rapidly. It is desirable to have the percentage of carbon below a reasonable figure for good ductility.

The maximum carbon content of the modern heat resisting alloys is approximately 0.70 per cent for castings and 0.20 per cent for sheet. Carbon is usually increased as the nickel content is increased.

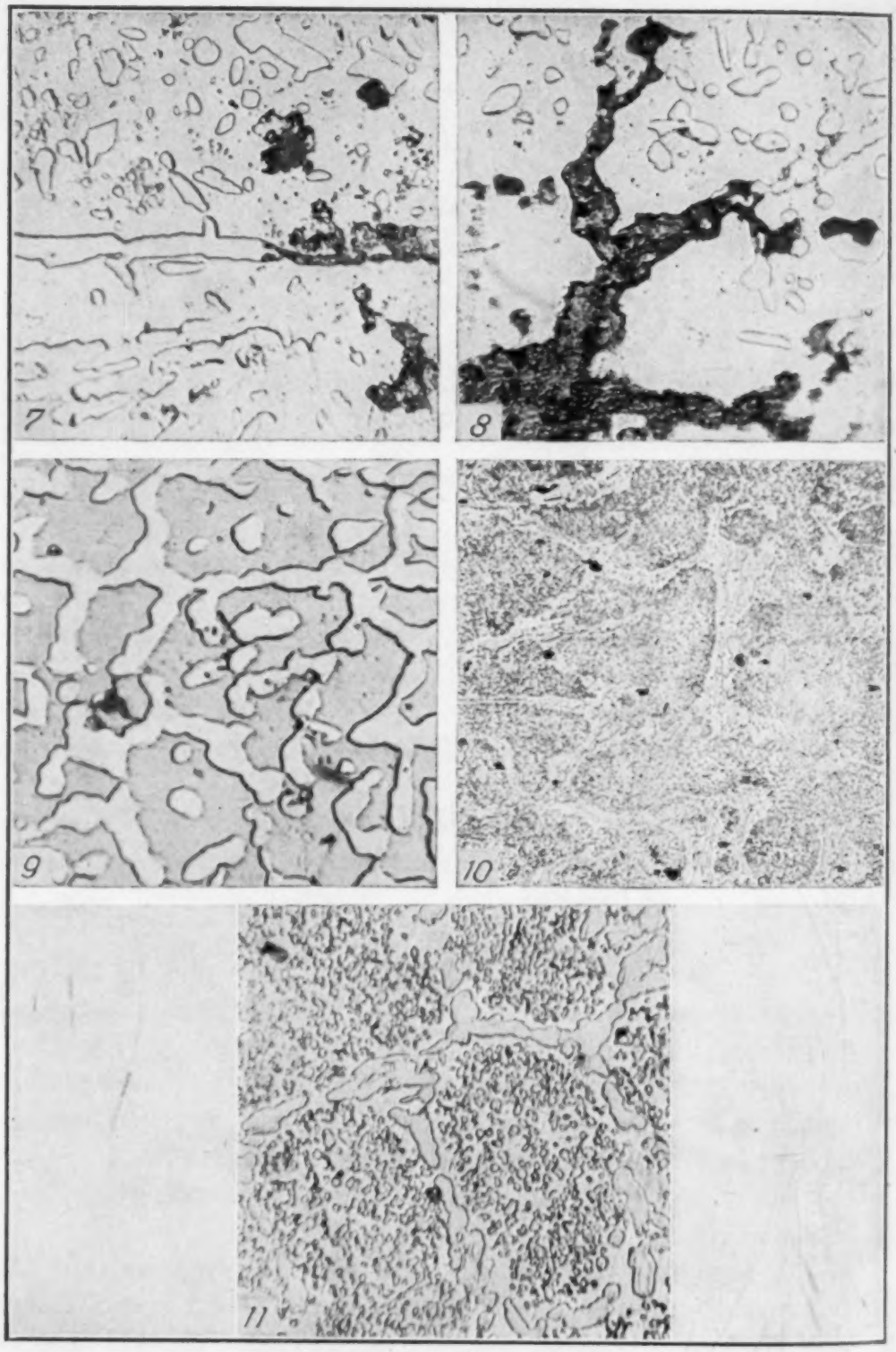
Various alloys of copper and iron have been tried but to no advantage. Aluminum and iron have been tried in various proportions and extensive investigations made on other low-priced alloys in the hope of finding something less expensive than the nickel-chromium, but without success.

Cast and sheet steel containers, coated by means of metal spraying, have shown possibilities but require further development. When coated with copper, carburization is stopped, while an aluminum coating improves heat resisting properties.

Of the alloys listed in Table I, Nos. 7, 8, 9 and 10 are used extensively as castings, and Nos. 3, 4, 5, 7, 9 and 10 for sheet.

The crystalline structure of these alloys is austenitic. The silicon and nickel, together with part of the chromium, are in solid solution in the iron and the excess chromium in the form of chromium carbide. The carbide often forms an incomplete network and in some cases finely divided irregular masses, depending upon the casting practice and cooling rate.

After long exposure to carburizing temperatures, oxidation and other forms of corrosion have been found to follow the carbide network, extending back from the exposed surface. The photomicrographs shown in Figs. 7 and 8, show oxidation following carbide particles from the surface inward. The carbide itself or the solid solution immediately around it, which is deficient in chromium, is



Figs. 7 and 8—Photomicrographs Showing Oxidation Following Carbide Particles from the Surface Inward.

Fig. 9—Photomicrograph Showing Normal Structure of the Cast Alloy.

Figs. 10 and 11—Photomicrographs Showing Sections After Being Subjected to Carburizing Atmospheres for Extended Periods. All Photomicrographs × 500.

oxidized first. These photomicrographs were taken at 500 magnifications at a point just beneath the surface of a container made of alloy No. 7, after 5000 hours service.

The normal structure of the cast alloy is shown in Fig. 9. Figs. 10 and 11 show sections after being subjected to carburizing atmospheres for extended periods. All were etched in Merica's reagent and magnified 500 times.

Physical Properties

Physical properties are naturally important factors in any steel or alloy. However, present foundry practice and casting limitations provide factors of safety which adequately meet the strength requirements of carburizing containers. An alloy exhibiting the best physical properties at normal temperature may have the poorest at carburizing temperature. An alloy having poor heat and corrosion resisting properties is soon reduced in section and weakened to an extent that will cause early physical failure. Actual strength in pounds per square inch is, therefore, not the most important factor.

As previously stated, the chief function of chromium in these alloys is its high heat resisting property. Silicon has a weakening effect at high temperatures. Nickel and carbon have a strengthening effect at both normal and high temperatures, without serious losses in ductility.

Some approximate results of physical tests of cast alloy are shown in Table II. The room temperature strength of these alloys

Table II

Alloy No. Table I	Ni	Cr	Room Temperature Ten. Str. p.s.i.	Yield Point p.s.i.	Elong. Per Cent	Red. in Area Per Cent	1600° F. Short Time Ten. Str. p.s.i.	Elong. Per Cent	Red. in Area Per Cent
7	35	15	65,000	45,000	10	10	20,000	25	35
10	65	15	65,000	45,000	10	10	20,000	25	35
9	60	12	75,000	50,000	30	40	30,000–40,000	30	40
3	12	24	85,000	58,000	18	15	20,000	20	30

Hardness averages 150 Brinell as cast.
Hot tests made at 1600 degrees Fahr. at 0.05 inch per hour.

after extended service is increased by carburization, but decreased by oxidation. Sheet alloys show a higher room temperature strength according to their hardness and amount of rolling. At higher temperatures, all physical properties are reduced rapidly. An alloy hav-

ing a strength of 50,000 pounds per square inch at room temperature may have less than 1000 pounds per square inch at 2000 degrees Fahr.

Recommended design, strength and thermal expansion data are shown in an article by J. D. Corfield, on page 382 of the A.S.M Handbook.

Reference is also made to a paper by N. B. Pilling and Robert Worthington published by the American Society for Testing Materials and the American Society of Mechanical Engineers in a joint symposium on the "Effect of Temperature on the Properties of Metals" in 1931. Page 495.

Container Life—Failures and Repairs

The life of any container, whether casting, sheet, or combination sheet and casting, depends mainly on design, composition and soundness of the alloy, care used in fabrication and welding in the case of sheet and combination containers, furnace equipment, type of fuel, operating temperature and the method and rate of heating and cooling. Most any foundryman can enumerate a hundred reasons why carburizing containers fail and can often trace the cause of a premature failure to a particular foundry operation.

Some of the common types of failures are briefly classified as casting failures, chemical failures and mechanical failures.

Casting Failures—Many failures are started from shrinkage cavities or voids, formed in heavy sections or abrupt changes in section where the cooling rate has not been uniform. Gradual changes in section from heavy to light and more uniform cooling rate by the use of proper risers on the light sections and chills on the heavy sections usually serve as good preventatives for these defects. Large cavities reduce the section and offer little resistance to thermal stresses and result in cracks. Small widely distributed shrinkage cavities, gaseous metal having blow holes, sand and slag inclusions, and segregation or dissociation of alloy into a mechanical mixture of component parts of alloy, all reduce the strength of a casting and allow leakage of gas at carburizing temperatures. These casting defects are almost always invisible from the surface of a casting and are only detected by the use of X-rays or destructive testing.

Cold shuts, or laps, are sometimes formed on the surface of a

casting, appearing as small smooth cracks. They are caused by too rapid cooling of the surface and as they are discontinuities in the metal surface, they reduce the strength, and if very deep, will collect oxide corrosion products and carbonaceous deposits which prevent contraction and cause cracks.

Chemical Failures—All energizers used in the manufacture of solid carburizing compound have a deteriorating effect on containers, regardless of the composition of the alloy. This holds true especially at the junction of the cover and the top of the container, where a flame temperature is produced which is higher than the carburizing temperature, and also at any defective areas at which gas leaks have formed. A gas leak, at the cover line or at a defect, once started, grows rapidly, burns away the surrounding metal and forms an opening of increasing dimensions. The protective oxide coating is destroyed, another is formed and destroyed, accelerating the disintegration of the alloy. This is due to high local flame temperatures and a fluxing action of the gases formed by the energizers, of which sodium carbonate is the most damaging.

Of equal importance with the composition is the cleanliness of the compound, since dusty fines permitted to remain within the compound will carry a very high percentage of energizer with a corresponding increase in the fluxing action referred to above. Since these fines are formed mainly by mechanical handling, any reduction in such handling will contribute to a lengthening of container life.

There are three important fuels used for heating carburizing furnaces; oil, gas (natural and artificial), and electricity. The fuel oils in common use generally contain a small amount of sulphur, the products of which when combined with the gases from the container, are very corrosive to alloy.

Some fuel oils have also been found to contain small percentages of chlorine, which is more active than sulphur. Further general deterioration is more noticeable in oil-fired furnaces than in those heated with gas or electricity, no matter what type of alloy is used in the containers. It has been proven by production tests that electrically heated furnaces are, on the whole, more favorable to long container life than either of the other types. From an economic standpoint, it can be stated that for a given operation the electric furnace is generally more expensive than the other two types, but considering the increased container life obtained, it may (under

favorable conditions) prove to be fully as economical as the oil or gas fired furnaces.

Mechanical Failures—Containers must be designed and made sufficiently rigid, and must be adequately supported to carry the load for which they are intended. Overloading causes bulging, sagging and eventually cracking. Thermal stresses concentrated in small areas should be avoided. Severe and unequal temperature changes causing one part of the container to expand or contract more rapidly than another, contribute to many failures. Round containers are more ideal where their application is practical.

Rough handling, either by men or mechanical devices, will contribute materially to container failures. Containers made from sheet or thin alloy castings are especially susceptible to failure from this cause. The chilling action of the heavy tongs of handling devices in contact with the hot container causes severe concentration of stresses.

The impact strength of the alloy is reduced as the container is used, and the strength of the alloy at carburizing temperature is greatly reduced.

In the two types of furnaces generally used for pack carburizing, the batch type and the continuous type, the latter contributes much to the lengthening of the container life. There is more uniform support on the roller rails of the continuous furnace than that effected by the uneven brick hearth of the average batch type furnace; the spacing of containers in the continuous furnace is definitely determined by the tray dimensions; and the continuous furnace furnishes a more uniform heating with a resulting decrease in the strain on the container.

Although the container life is better now than it ever has been, information gained through the careful study of the causes of failures will lead to greater improvements in material, design and manufacture.

Repairs

Many cast and sheet containers, after they have given good service, are discarded on account of some defect, which, although having made them unfit for economical service, has not entirely destroyed their usefulness. A minor repair may recondition them and allow 50 per cent additional useful life. When properly heated, most any container, cover, or tray can be straightened and formed to its original shape.

Cracks and porous sections can be welded, restoring the original strength to the container and rendering it reasonably leak proof. The welding of thin sheet is much more difficult than the welding of a casting. The welding of a sheet to a casting, especially when both have absorbed considerable carbon, demands the best workmanship. Skillful welding technique is a major requirement. The material must be cleaned by chipping, grinding or machining, and all oxide

Fig. 12—Photograph Showing the Manner in which a Large Oblong Casting Will Deform with Continued Service.

coating removed from the portion to be welded. Acetylene welding is usually employed in such repairing, the welding rod being the same composition as the parent metal. Many good repairs have been made on cast and sheet containers by welding a sheet alloy patch over the defect.

Many containers after a second repair have shown long service. It is best to make repairs when defects are first noticed, as small cracks or porous sections cause rapid disintegration of the alloy.

Fig. 12 shows how a large oblong casting will deform with continued service. The closing in and cracks around the top are due to heating and cooling strains. Such a casting can be hot straightened and welded and will continue to give good service, as it has nearly all of its original weight.

Container-Furnace Relation

The relation of container design to furnace design, when properly planned and executed, will effect an appreciable saving in time, fuel, alloy life, and floor space, and reduce substantially the total investment. In liquid and gas carburizing, continuous or batch type, where containers are not removed from the furnace, this relationship is ideal as the two units are designed and built as one. While this is not possible with solid compound carburizing, where containers are repeatedly passed in and out of the furnace, the two units can be designed to give, through their combined function, service comparable to the built-in container.

The nearer the approach to absolute uniformity of heating in any type of furnace, the more simple will be the problem of carburizing and the less complicated the container design. Because of the employment of the counterflow principle, electric heat was very popular several years ago, and excellent records were obtained from containers used in these furnaces. However, it is significant that with this experience there were fuel fired furnaces operating likewise on a counterflow principle which produced performance records of equal merit.

The advent of direct quenching has changed the furnace designs rather materially, and has been accompanied by certain necessary improvements in container design which have approached the maximum of economy reasonably to be expected.

Electric furnaces, especially those of great width in comparison to their length, if the heating elements are not correctly placed, often produce variable results, overheating the containers nearest the elements before those in the center of the furnace reach the carburizing temperature.

Fuel-fired furnaces of similar proportions having a multiplicity of small burners, well distributed, reduce the tendency toward unequal

heating by reason of a more complete circulation of furnace atmospheres throughout the heating chambers.

Continuous furnaces having suitable heating zones, afford a heating cycle which is advantageous not only to alloy containers but to work being carburized.

In spite of the many modern installations, there are many carburizing furnaces in use today showing some temperature variations, which if not carefully controlled, have their undesirable effect upon the efficiency and life of the alloy containers. Various designs of containers have been used to compensate for these unfavorable heating conditions, light and heavy sections, corrugated sections and other variations to give strength and flexibility where stresses are imposed by temperature differentials.

Covers for inverted containers, and trays for upright containers, for use on skid conveyors or roller rails of continuous furnaces, are equipped with guides adapted to the rail spacing, and must be designed to carry compression or thrust loads as well as the loaded containers. Hinged, jointed trays are used in some applications to meet unfavorable furnace conditions, and have proven a simple method of breaking up the span of one long beam into one or more short beams, in passing over the rails. In some cases the weight of trays has been reduced as much as 40 per cent by this design, while the life has been increased. Types of conveyors made to move containers through furnaces without permitting compression of the tray will permit the designer to design the tray to carry the load rather than to resist thrust.

Intelligently conceived handling methods employed at the discharge end of continuous furnaces, particularly in direct quenching, have done much to lengthen container life. Suitable equipment for this purpose should be considered a necessary part of any carburizing installation.

The overloading and crowding of containers in furnaces not only reduces the container life, but the quality of work done. Methods used to speed up or increase the rate of carbon penetration such as high temperature and very active compounds are costly from the standpoint of container life, but are sometimes necessary and justified when production requirements, for short periods, are beyond the capacity of the equipment at hand.

Furnace designers give little consideration to containers other than overall dimensions and total weight. The furnace lining plays

an important part in the performance of containers. Insulating refractory, absorbing less than 5 per cent of the heat input, does not have the equalizing effect of standard fire brick. This is very noticeable in the long alloy muffles of continuous gas carburizing furnaces. With the insulating refractory, higher temperatures are noticeable in sections immediately adjacent to the burners, while in furnaces lined with standard firebrick having high heat absorption, more equal muffle temperatures are realized. To a considerable extent, these same conditions are experienced in furnaces for liquid carburizing when fuel firing is used. The most modern containers for liquid baths, however, make use of immersion heaters placed inside the bath. This method of heating does not restrict the designer to the use of high grade alloy nor thin wall sections. The containers can be used in direct contact with the furnace lining, permitting their full support and resulting in long life. Flame impingement on any container is disastrous to alloy, and seriously impairs the quality of work.

If the importance of the furnace-container relationship can be fully realized by designers of this equipment, greater production, higher quality and lower costs will result.

Work-Container Relation

The lack of organized data on the relationship of work to the container is in a great measure due to the three following factors—

1. The difference of opinion concerning the composition of the carburizer and the amount necessary.
2. The assumption that when a good design has been worked out for one particular part, it will be equally efficient for a number of different parts.
3. The objection to increasing the number of containers beyond the absolute minimum required.

However, at present it is the consensus of opinion among authorities on the subject of carburizing, that the most efficient operation can be obtained only when the proper relationship between the part to be carburized and the design of the container has been established. In production carburizing, an essential part of this relation is the ratio of the weight of the work to the weight of the loaded container. Furnace manufacturers take this into account when submitting proposals for carburizing equipment. All of their figures and quotations are based on the gross weight to be handled per hour

by the furnace and not on the net weight of parts to be carburized, because of the difference in carburizing practice.

Early investigators of pack carburizing determined, by experiment, that any change resulting in a reduction of the time cycle required to produce a given case depth (particularly that portion referred to as the heating-up period), would increase the efficiency and decrease the cost of operation. Later, as changing conditions demanded revisions of existing carburizing practices, further experiment showed that maintaining a close container-work relationship

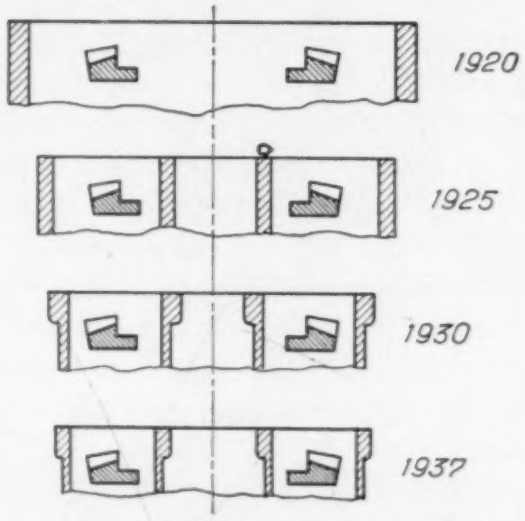


Fig. 13—Sketch Illustrating Relative Size and the General Trend in Cylindrical Ring Gear Pots.

was the most effective means of reducing the heating-up portion of the carburizing cycle. With containers conforming to some extent to the size and shape of the part or group of parts, less compound was needed and even further economy could be realized.

At the time that direct box quenching was considered, some years ago, the importance of work-container relationship was definitely established through the results obtained by carburizing a number of like parts under the same conditions in containers of different size, shape, and wall thickness. In a series of tests conducted to determine the best possible type of container for carburizing automotive ring gears, it was learned that square and rectangular boxes were entirely out of the question because a temperature differential existed in the gears themselves, due to the fact that the container shape did not conform to the shape of the parts. This resulted in excessive distortion. Moreover, the amount of compound needed for these types of

containers was greatly in excess of the actual requirements. These tests also proved that in cylindrical containers of the same wall thickness, any decrease in the diameter of the pot—not exceeding the safe limit where parts may be easily removed while hot, without damage—will show a reduction in weight, compound used, and a corresponding reduction in the carburizing cycle. It was further noted that gears carburized in containers with straight-through chimneys required less compound and less time when compared with the same parts carburized in containers of the same metal section but possessing no chimneys.

It can be stated further that containers made from sheet or thin cast heat resisting material will show a greater efficiency than boxes of relatively heavy metal section. In most cases, if the containers are properly designed to meet the requirements of a particular application, a reduction in the carburizer container cost can be realized. Where carburizing practice will permit a reduction in the usual ½ or ⅜ inch cast container wall thickness to ⅛ or $\frac{3}{16}$ inch metal section, the reduction in the weight of the box will increase the net weight-gross weight ratio.

Weight of cast container and cover reduced from approximately 300 to 100 pounds.

Weight of compound reduced from 40 to 25 pounds.

Heating up period reduced 3 to 4 hours.

Total time for 0.060 inch case reduced from 18 to 14 hours.

Number of gears carburized in one batch type furnace load increased from 142 to 162.

The sketch in Fig. 13 illustrates the relative size and the general trend in cylindrical ring gear pots.

Work-Compound-Container Relation

Although modern carburizing compounds have been refined, they are still composed mainly of coke and charcoal. These materials are very poor conductors of heat. Therefore, the thinner the layer of carburizing compound between the outer surface of the part or stack of parts and the container wall, the greater will be the reduction in the heating-up portion of the carburizing cycle, with a corresponding reduction in fuel consumption. In the past, where the available compounds produced very nonuniform case depths, the assumption that relatively large amounts of compound were required to properly car-

burize the work was somewhat justified. Today, with the improved compound manufacturing technique, where the compound is in contact with the work, excellent results can be obtained with a surprisingly small amount of material. Several years of experience have proved that, when pot cooling is the practice and the containers conform to the shape of the part or stack of parts whose entire surface is to be carburized, the layer of compound between the part and the container wall can be as small as ¼ inch without impairing the quality of the case or increasing the overall carburizing cycle. Where parts are quenched direct from the carburizing temperature, and particularly those requiring individual quenching, in many cases, this compound layer must be increased slightly to permit handling while hot, without damage. Where this close relation exists, the tendency of the compound to ash is also reduced because there is less compound exposed to the air.

Production experience has further proven that while the weight of the part or group of parts controls to some extent the amount of carburizing material in contact with it during the period required to produce the desired case depth, the area of the part or parts is also a controlling factor in the amount of compound required.

There should be a distinction made between the amount of compound actually required to produce the desired type and depth of case and the amount actually used. In the case of parts requiring selective carburizing, it has been found that where the container definitely conforms to the shape of the part, and the ¼ inch compound layer can be maintained, an approximate ratio of 0.01 pound to 1 square inch will produce the desired case depth, with a corresponding reduction in the time required.

Further investigation proved that, regardless of some theories to the contrary, no difference in the carburizing cycle occurred when cylindrical containers loaded with six ring gears were run, first, with a closely fitting cover in place; second, without any cover whatever; and third, with a 50-pound weight resting on the cover. In the second instance where no cover was placed on the container, the compound ashed to a depth of about two inches. Otherwise, no difference was noted between these three test runs, in either depth or type of case on the gears, as shown by micro-examination. In the third instance where a weight was placed on the cover of the container, the effect was identical to that obtained by inverting the container. Although some operators still consider that this inverting

of the container increases the pressure of the gas, and hence speeds up carbon penetration, actual investigation with the weighted cover has proven this idea false.

Container Design

For a satisfactory and permanent solution to any carburizing problem, a careful study must be made of all the essential factors involved; such as the size, shape, weight and composition of the part, the type of container, available furnace equipment, fuel for heating, analysis and amount of compound required, and operating temperature. Perhaps the most important of these is the container design.

Although there has been a tremendous advance in the composition and casting of heat resisting alloys, as well as in welding, rolling and fabricating technique, container design has not taken full advantage of these improvements. At present, the containers in general use do not embody a close furnace-container relationship and work-container relationship; this latter factor in the case of pack carburizing, determining the amount of carburizer used.

Where liquid baths constitute the carburizing medium, and batch loading is the practice, there is a definite work-container relationship. The size of the load is determined by the amount of molten salts contained in the pot. Most salts are of such a nature that whenever mass loading is great enough to lower the bath temperature below certain limits, the carburizing efficiency of the bath is impaired. Since commercial salts show a considerable variation in composition, information on safe operating temperature limits should be obtained from the manufacturer supplying the material.

The life of the container depends mainly on wall thickness, fuel used, furnace equipment, mechanical strength and soundness due to casting practice, and composition of the alloy.

In batch type gas carburizing where the retort revolves, container design is to a great extent predetermined by the furnace design. Parts which do not lend themselves to carburizing in this manner can be carburized on racks designed to provide smooth flowing contact between the gas and the parts in the muffle type of batch gas carburizing furnace. Muffles for this type of furnace are designed to meet individual carburizing problems. In a few of the larger plants where there is steady output of a great number of similar parts requiring similar types and depths of case, carburizing in continuous

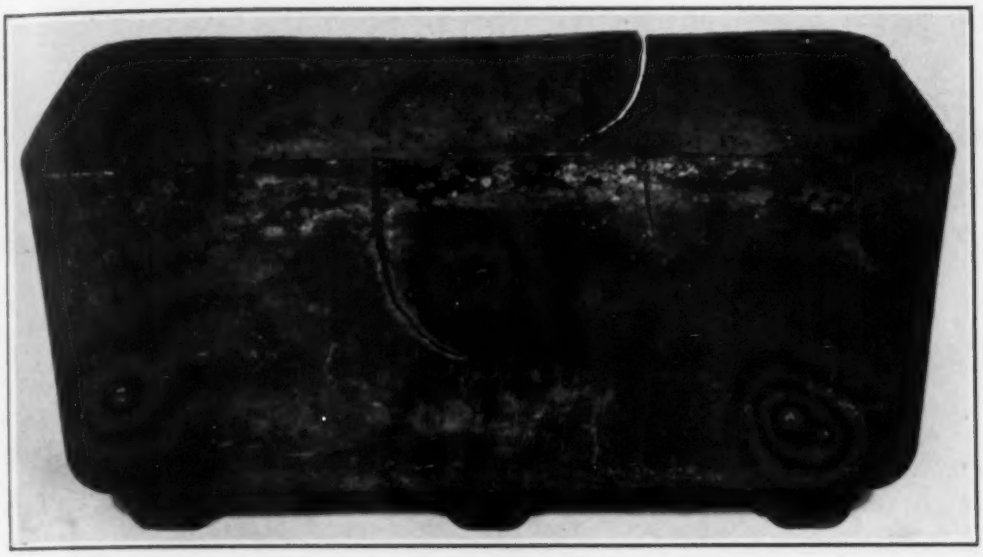


Fig. 14—All-Cast Container Made from Alloy No. 7, Table I, of ⅜-inch Metal Section with Inside Dimensions; Length 29 Inches, Width 11 Inches, and Depth 13 Inches, With No Reinforcing Band at the Top, and Supported by Six Hollow Legs 2½ Inches in Height, Cast Integral. Weight 166 Pounds.

Fig. 15—Bottom and Side View of Fig. 14.

gas furnaces is replacing pot carburizing in continuous furnaces. Where this condition regarding the parts exists, and direct quenching is the practice, carburizing in continuous gas furnaces will produce more uniform results at a lower price per piece than will pot carburizing in either the batch type or continuous furnaces.

With the introduction of the continuous gas carburizing furnace, new problems in container design originated, because designers of

Fig. 16—Combination Sheet and Cast Container with Cast Base of ½-Inch Metal Section Made of Alloy No. 7, Table I, Loosely Attached to Heat Resisting Sheet Body (Alloy No. 5, Table I), of ⅜-Inch Metal Section. (Same Inside Dimensions as Fig. 14.)

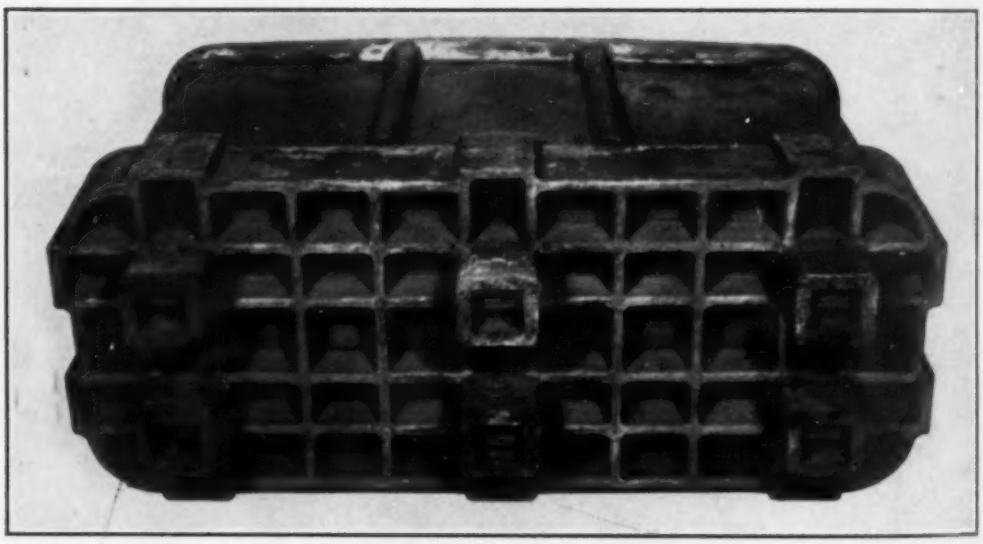


Fig. 17—Side and Bottom View of Fig. 16.

alloy equipment had never before been called upon to produce muffles of from 19 feet to 27 feet in length, which were exposed to oxidation and carbon penetration at temperatures ranging from 1650 to 1800 degrees Fahr. After five years of production experience, a few of the important factors governing muffle design have been found to be:

1. The size and weight of the part or parts, and the production requirements.

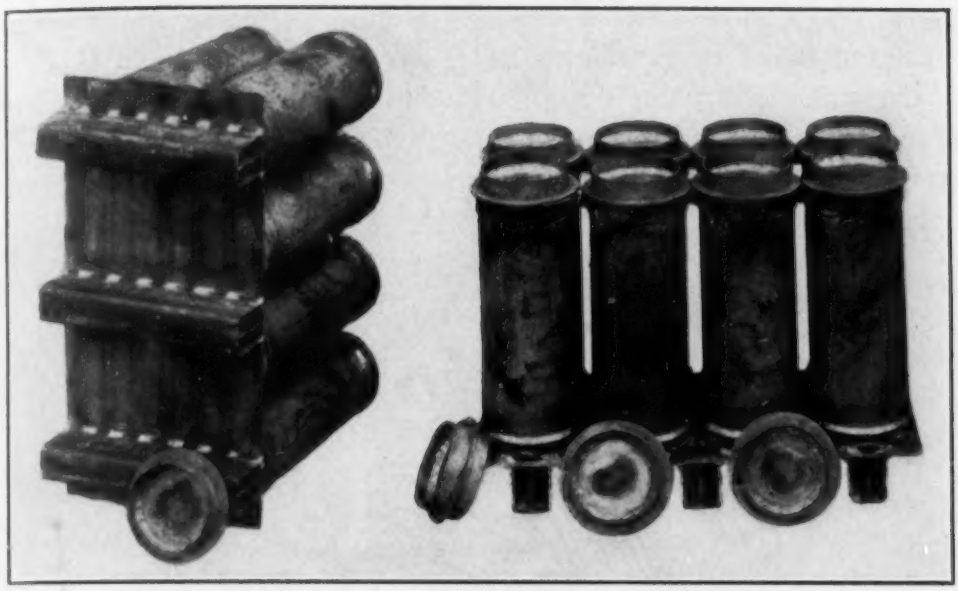


Fig. 18—This Unit Consists of Eight Cylinders of ⅛-Inch Heat Resisting Sheet Metal (Alloy No. 5, Table I) Spaced 1 Inch Apart and Attached to a ⅛-Inch Corrugated Sheet Base Supported by Three Reinforced U-Shaped Legs of ⅛-Inch Metal Section. The Cylinders are 3½ Inches in Diameter and 15 Inches High (Inside), and Are Reinforced at Top with a ⅛-Inch x 1-Inch Band Welded in Position. Covers are of the Reversible Type and of ⅛-Inch Metal Section.

2. The relation between the dimensions of the muffle and the number of trays to be pushed through per hour.

3. The proper placing and size of the corrugations at the top of the muffle to prevent distortion.

4. The method of joining the various cast sections, comprising the muffle into a single unit.

5. The relation of the cross sectional area of the muffle to the cross sectional area of a loaded tray.

The authors have had five years of production experience with a continuous gas carburizing furnace having a muffle length of 19 feet, a cross sectional area of 328 square inch, an operating cycle of one tray every 30 minutes, an operating temperature of 1750 degrees Fahr., and a present gross load per tray of 238.5 pounds (this includes six gears of 21 pounds each, spacers of 1 pound each, and 1 tray of 64.5 pounds). To date, the best muffle design has been found to require the following points:

1. No flanges except at each end where the muffle proper joins the gate casting.

2. Three-inch corrugations about two inches deep, properly spaced.

3. ½ inch metal section

4. All muffle sections joined by atomic hydrogen or gas welding on the outside at beveled joints.

In considering the design of containers for pack carburizing, the most important factors are size, shape and weight of the part or parts, the combined experience of alloy casting manufacturers, fabri-

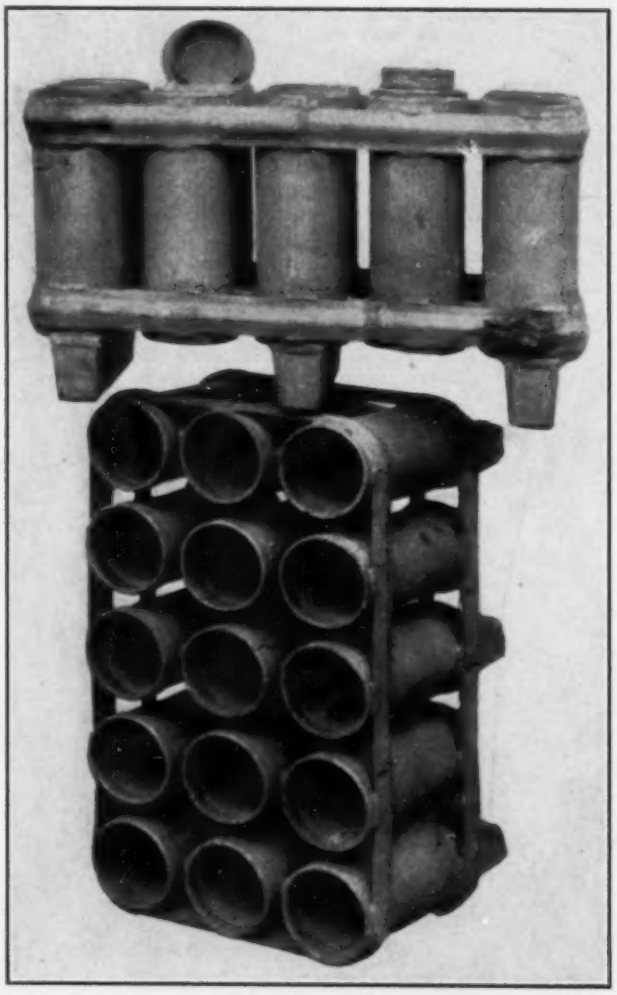


Fig. 19—This 15 Cylinder Container Was Made Entirely of ⅛-Inch Heat Resisting Alloy Sheet (Alloy No. 5, Table I), and Assembled by Gas Welding.

cators of sheet containers and of the carburizing department using the containers, and an alloy metallurgically suited to the operation and mechanically sound, which is intelligently related to the service requirements and to the furnace equipment available.

Wherever possible, cylindrical containers, either with or without chimneys, should be employed, because they show a greater number of heat hours than square and rectangular containers of like metal

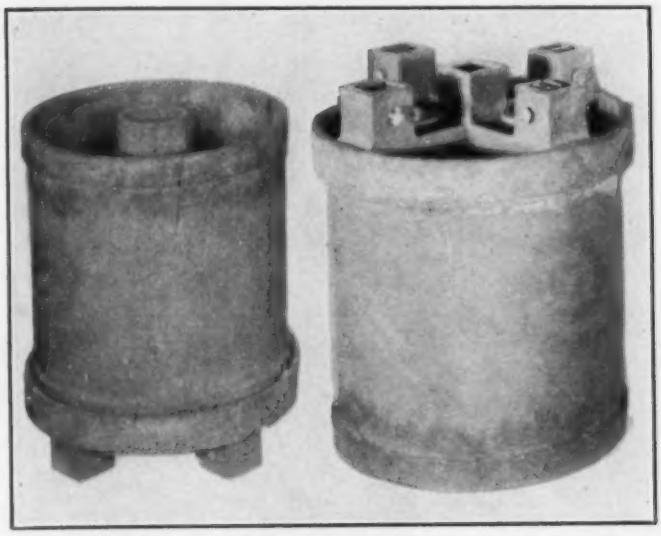


Fig. 20—Container Body is Made of ⅛-Inch Heat Resisting Alloy Sheet (Alloy No. 5, Table I), 15 Inches High and 14 Inches Diameter (Inside), With 4-Inch O.D. Chimney. Strength is Added to the Top of Chimney and Pot Body by a ⅛-Inch x 1½-Inch Reinforcing Band. The Base is ⅜-Inch Cast Alloy No. 7, Table I, and is Loosely Attached to the Container Body by Means of Eight L-Shaped Pieces, 1 Inch Wide.

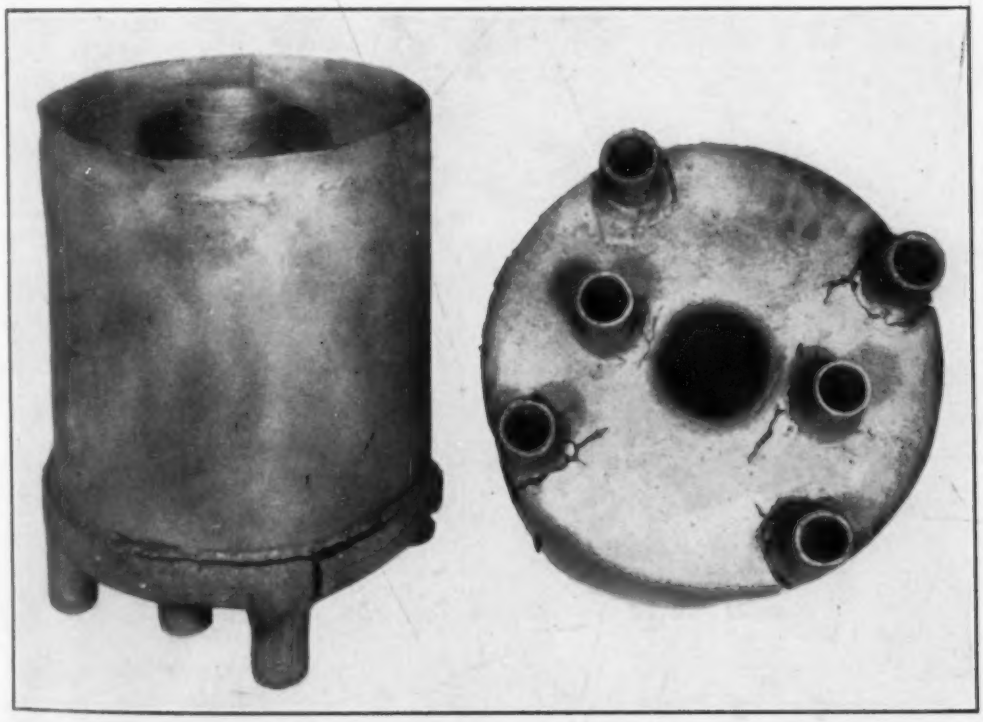


Fig. 21—The Dimensions of the Container Illustrated in Figs. 21 and 22 are Identical Except the Base Casting and Design. In This Case, the ⅛-Inch Metal Section of the Container Body was Gas Welded to the ⅜-Inch Cast Base. (Alloy No. 7, Table I).

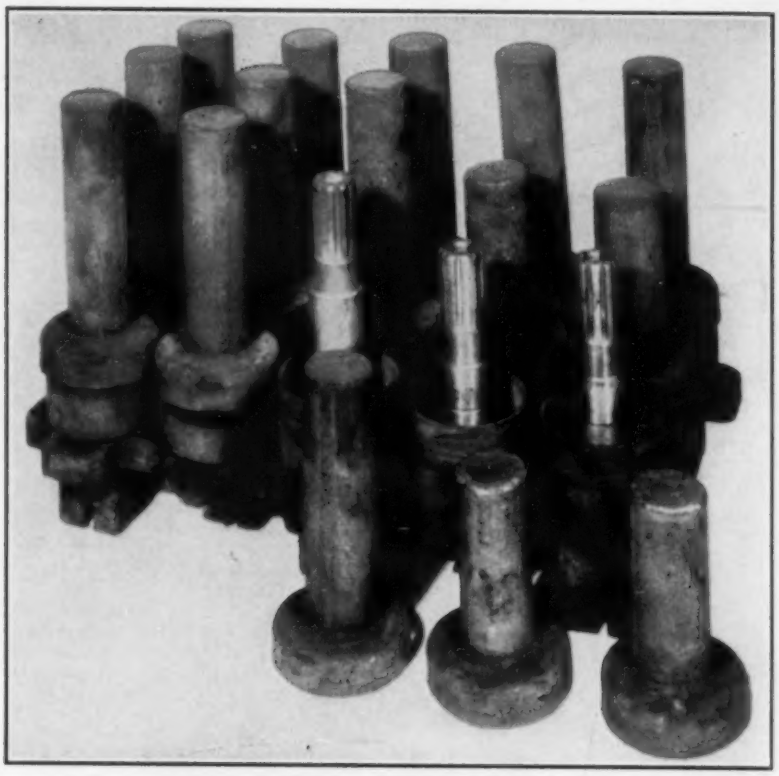


Fig. 22—Photograph illustrating a Container that Definitely Conforms to the Shape of the Part.

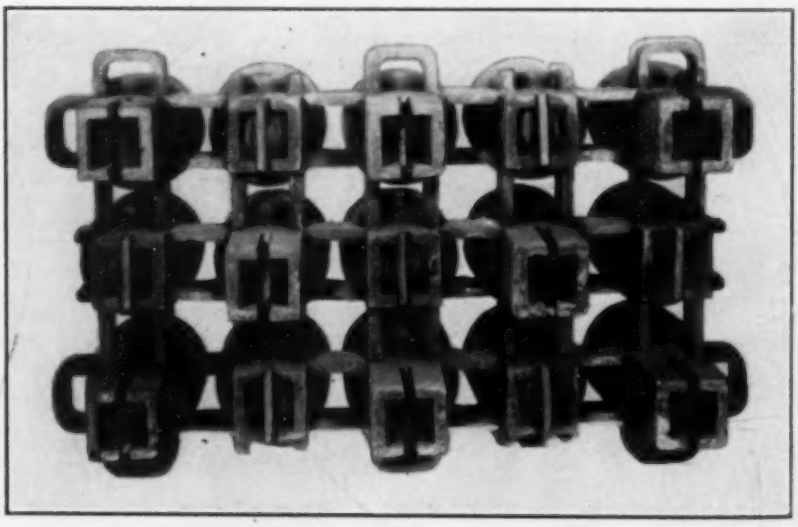


Fig. 23—Photograph Showing Base and Assembly Details of Fig. 22.

section and under the same operating conditions. Where the carburizing equipment must include rectangular containers for special purposes, greater economy will result from using a sheet metal con-

tainer loosely attached to a cast base with legs, and properly reinforced, than from using an all cast container. This is shown in photographs in Figs. 14, 15, 16 and 17.

Even in plants where the greater portion of production consists of relatively long parts, such as automotive drive worms and pinions, brake camshafts, etc., and continuous gas carburizing cannot be considered, they can be carburized more uniformly and with less distortion in a vertical position in cylindrical containers.

The performance of the container shown in Fig. 18 was unsatisfactory. Distortion due to nonuniform heating caused premature failure and resulted in the modified design shown in Fig. 19.

The cylinders were held in space relation by ¼ x 1 inch metal spacers and two V-shaped reinforcing bands 3 inches wide, completely surrounding the unit at the top and bottom. Enclosed legs of a modified U-shape supported the container. No bottom was required, allowing a maximum of convection heating. Further development of the principles embodied in this type of container (Figs. 18 and 19), and their intelligent relation to the particular application, will show decided advantages over the conventional methods usually employed.

Whenever possible, carburizing of parts in individual containers or groups of these units mounted on suitable bases should be the practice, because there is less distortion, more uniform heating, less compound per piece required, and a reduction of the carburizing cycle over that obtained with mass carburizing of the same parts and under the same conditions.

Where the advantage of containers made from sheet or thin walled castings is to be retained under comparatively severe service conditions, such as in batch type carburizing where the container design must incorporate an arrangement of legs, experience has shown that a container loosely mounted on a relatively heavy cast base is more efficient than a container having its base cast integral with it or welded to it. Fig. 20 illustrates this type of design and assembly.

There are two obvious reasons for this. If there is a difference in the composition of the container and the base, and these are rigidly attached by welding or other means, the difference in expansion between the base and container causes premature failure at the junction. The massing of metal due to assembly practice causes a very abrupt change in thickness of metal sections, resulting in dis-

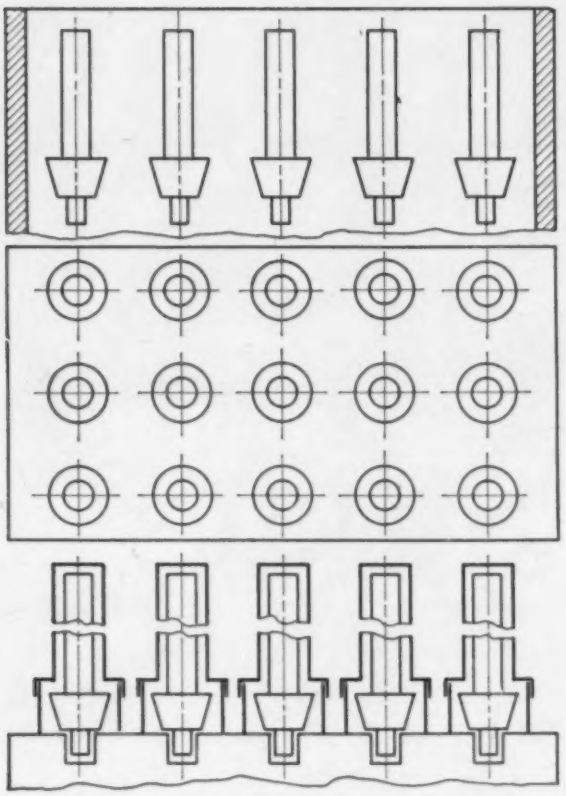


Fig. 24—Sketch Illustrating the Relation Between Rear Axle Pinions Packed in an Oblong Box and in the Individual Cups.

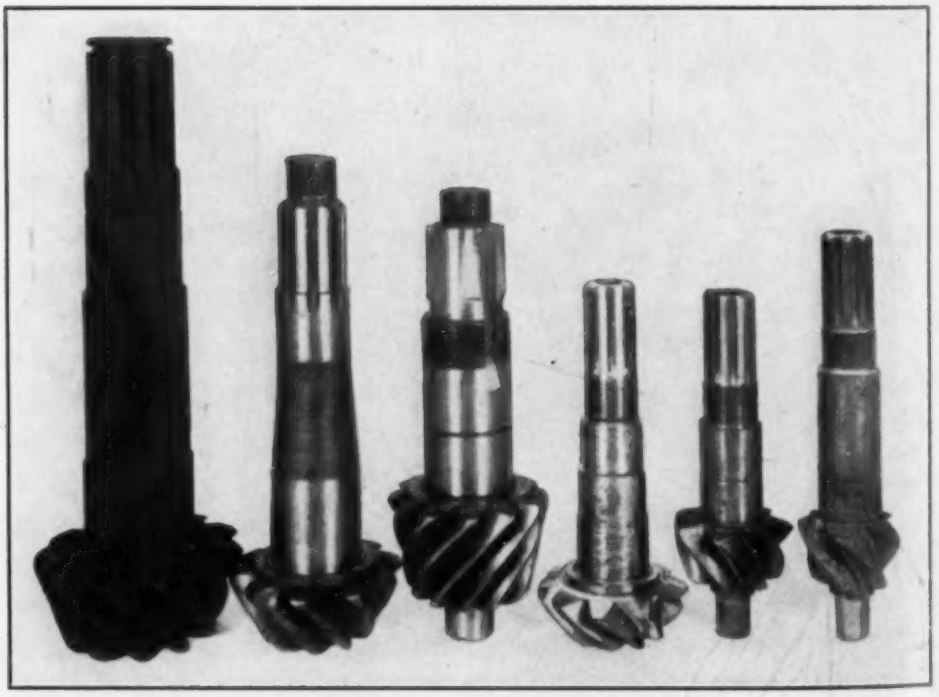


Fig. 25—Photograph Illustrating a Group of Pinions of the Keyway and Threaded Type of Shaft.

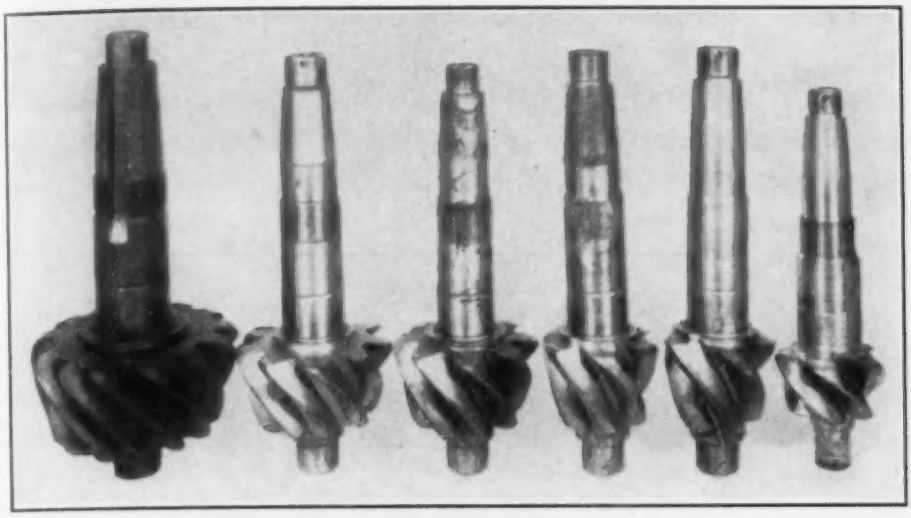


Fig. 26—The Largest Head Diameter Shown Here is 6¼ Inches, Body Diameter of 2¼ Inches, and a Length of 10¼ Inches.

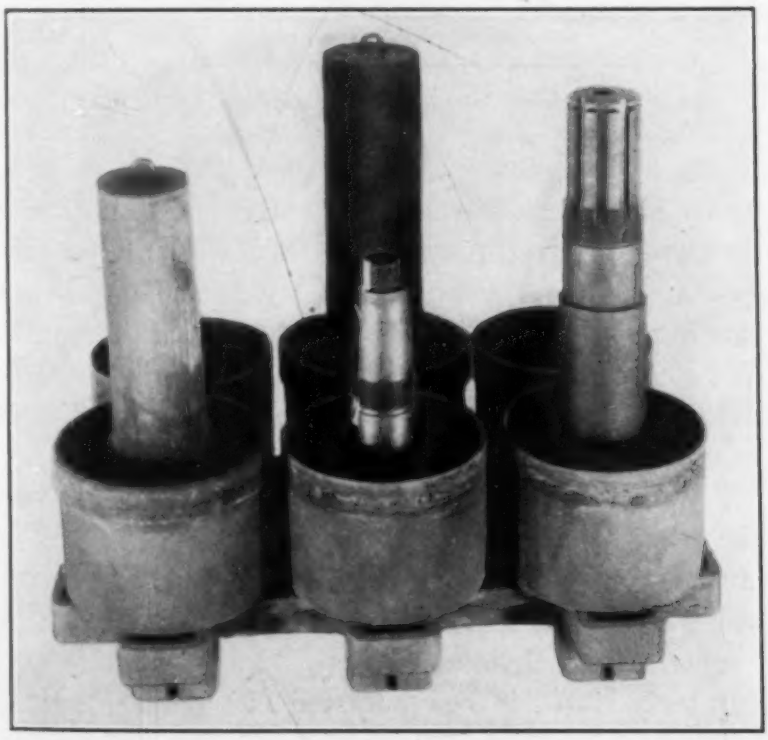


Fig. 27—This Design Shows Six Cup and Thimble Assemblies, Loosely Attached to Cast Bases as Shown in Figs. 22 and 23. This Design is Suitable For the Largest Type of Pinion Illustrated in Figs. 25 and 26. The Cups are 5 Inches High and 6⅛ Inches Diameter (Inside), Made of ⅛-Inch H.R.A. Sheet. The Larger of the Two Lids Shown is 3 Inches in Diameter, and 10 Inches Long. The Smaller is 2½ Inches in Diameter and 8 Inches Long.

tortion of the container. An example of this type of failure is shown in Fig. 21.

Another advantage of having the base loosely attached is that whereas the container is subjected to both oxidation and reduction corrosion, the base is exposed to oxidation alone. This means that

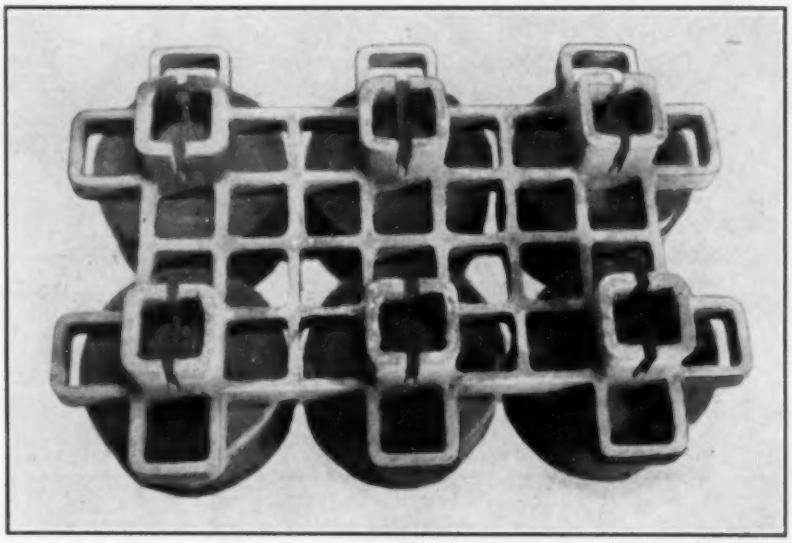


Fig. 28—Photograph Showing Details of Base Casting and Assembly.

under ordinary conditions one base will outlast several containers and being easily removed, need not be discarded with the containers.

In some applications where parts are carburized all over in the conventional manner and a portion is drawn back to approximately core hardness because of some particular requirement, specially adapted container design will eliminate the necessity of further operation by carburizing only the desired area.

Fig. 22 illustrates a container that definitely conforms to the shape of the part.

This unit consists of 15 cup and thimble assemblies of ⅛ inch H.R.A. sheet (Alloy No. 5, Table I), attached to a ⅜ inch cast 35-15 alloy base supported by 8 slotted legs.

The cup body is 3¾ inches diameter and 3 inches deep (inside), and the thimble is 1¼ inches diameter and 1⅝ inches deep. The thimble is attached to the cup by gas welding. The thimbles extend into the enclosures in the base casting spaced to provide ⅜ inch clearance between cup O.D. with the bottom of the cup resting on the top of the grid base. Assembly is made by welding ¼ x ⅜ inch

bar to the bottom of the thimble which extends to the bottom of the rib of the base and to the top of the slot in the legs.

The lids are made of ⅛ inch heat resisting sheet alloy (Alloy No. 6, Table I) and are $\frac{1}{16}$ inch larger than the cup diameter providing $\frac{1}{32}$ inch clearance when assembled in position. The lids are

Fig. 29—Photograph Showing Fixture in Position Before Filling With Carburizing Compound.

provided as shown, in two lengths (8 and 5½ inches) to suit requirements. Bumpers are cast on the side and ends of base to provide protection against damage to cups. Containers of this type can be made to suit parts of different size and weight, such as are shown in Fig. 23.

Fig. 24 illustrates the relation between rear axle pinions packed in an oblong box and in the individual cups. The oblong box packed with 15 pinions contains 45 pounds of compound, while the 15 individually packed cups, occupying the same furnace space, contain a total of 4.2 pounds of compound. The weight of the alloy is very nearly the same in each case.

Differences of as much as eight hours have been recorded in the heating-up time, when thermocouples were placed in a pinion in the center of the oblong box and one in a central cup.

Fig. 25 shows a group of automotive drive pinions of the spline type, varying in size, length and body diameter. The largest has an

overall length of 16 inches, a width at the head of 6⅛ inches, and shaft thickness of 2½ inches. The smallest pinion has a shaft thickness of 1⅞ inches, a length of 11 inches, and a head diameter of 2½ inches.

In selective carburizing, where subsequent operations or service requirements are such that certain portions of the part be left uncarburized and copper plating or the application of other types of protective coating are essential to insure against carbon penetration, container design, in many cases, will accomplish the desired result without the aid of further precaution.

The container and fixture shown in the accompanying photograph (Fig. 29), was designed primarily for the selective carburizing of automotive steering arm balls, and while detail revisions in design may be necessary through further development, results with the container, as shown, definitely prove its commercial value. The fixture consisted of a main plate and pilot plate of ⅛ inch heat resisting alloy sheet, held in space relation by four studs with an eye bolt to permit easy removal from the pot. It is interesting to note that no special precaution was necessary to insure a tight seal between the upper and lower compartments of the container during the carburizing operation. The cover conforms to the container and fixture requirements.

In container design, it is well to avoid metal sections greater than ½ inch, or in extreme cases, ⅝ inch, since the greater the thickness the more danger there is of failure due to shrinkage cavities, and other casting defects. There should be as few metal intersections as possible because, unless proper provision is made by the foundry, the two sections will tend to pull away from each other during cooling and this will result in a zone of weakness in the finished container.

Summary and Conclusions

Greatly improved carburizing results can be obtained by more scientific container design.

Time of carburizing can be reduced, as well as the weight of compound and alloy required, for a given amount of work.

It is the opinion of the authors that any advance in carburizing container design must center around the following fundamentals:

1. More definite planning and consideration of all relating factors by container manufacturers, furnace builders and all those responsible for the carburizing operations.

2. Further improvements in foundry practice and the production of sound, clean castings.

3. More widespread use of the X-ray for the inspection of castings.

4. Improved heating and handling conditions.

5. Improvement in the melting and rolling of heat resisting alloy sheet.

6. Further improvement in welding as applied to heat resisting alloys.

7. Intelligent relation of the container design to the shape of the parts to be carburized.

At present there is a wide variation in carburizing practice, resulting from differences of opinion as to the merits of various container designs, the optimum carburizing conditions, and the composition of alloy and carburizing media. In spite of this, it seems plausible to the authors that some standardization could be accomplished by establishing a kind of clearing house, such as a sub-committee of the A.S.M., for collecting all experiences, new ideas, and information from production tests relative to the subject.

The authors gratefully acknowledge the encouragement and assistance given by the following individuals and firms:

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DISCUSSION

Written Discussion: By H. W. McQuaid, metallurgist, Republic Steel Corp., Cleveland.

This is, to me, one of the most interesting papers which has been presented for a long time in the field of carburizing. Having been very intimately connected with the developments described in this paper, I, of course, have a strong interest in the subject. Mr. Roush and Mr. Dames are certainly to be complimented on this presentation and it is hoped that their discussion of the advantages to be obtained from a scientific design of carburizing boxes will be carefully considered by all interested in commercial carburizing.

There is one thing which I believe was not sufficiently stressed in the development of these sheet containers which might be of interest to those who have occasion to apply the thoughts expressed to other applications. It was

quite evident from the beginning that the success or failure of the sheet metal boxes, as described, depended primarily on the weld which was obtained. It was generally found that the failure of the sheet metal container was primarily due to some deficiency in the weld. Containers which were properly welded would give an excellent account of themselves and fail either by burning at the exposed top edge or by some local irregularity in the sheet metal itself.

It was soon found out in the design of these units consisting of a group of cylindrical boxes that it was extremely important that the very minimum of constraining tie to a given box should be permitted. A directional stress of any magnitude on heating and cooling to the carburizing temperature results in a change in shape of the box. Any ties or fastenings which tend to prevent unrestrained, uniform movement in all directions on heating or cooling will introduce stresses which soon result in a box which has lost its original shape in the endeavor to conform to the stresses applied. They should be so designed that as nearly as possible the boxes are loose and unsupported and, wherever possible, cylindrical in shape.

It seemed to me that the writers might have expressed a little more strongly the saving in time which can be obtained by reducing the thickness of the insulating compound around the work. In a box of the design shown in Fig. 19, for example, it has been possible to carburize steering gear worms to a depth of 0.045 inch and to quench them in an overall time of three hours and fifteen minutes. These parts were made from S.A.E. 4615 and the overall time compares more than favorably with gas carburizing.

Another point which the authors might have stressed more strongly from their experience was that of the beneficial results which are possible by the use of cylindrical containers such as they describe. These are particularly valuable in reducing the warpage of cylindrical parts such as pinions, worms, pins, etc., to a minimum. In a box of this type, such parts are carburized in a vertical position and the application of the heat is uniform in all directions. In removing them from the box for direct quenching, they are maintained in the vertical position which results in an important elimination of crooked stems, etc. In fact, it was for this purpose that the box shown in Fig. 18 was originally developed and the results as regards reduction of warpage and uniformity of case well justified the expense involved.

This paper is one which is of considerable commercial importance and represents the results of several years of very intensive effort, especially on the part of Mr. Dames. Much of the work was done under handicaps imposed in operating one of the largest and most complicated heat treating departments in the country and much of the work was done during the late, well known depression, when development work was difficult to justify.

It is recommended that all producers of carburized material give very careful consideration to the economy which can be developed by the use of scientifically designed carburizing boxes intended to reduce the insulating effect of the compound to a minimum and at the same time insure uniform heating and cooling.

Written Discussion: By S. L. Widrig, Spicer Mfg. Corp., Toledo, O.

Due to the similarity of work carburized by the authors and ourselves, we can well appreciate the value of such a paper. Carburizing is an expen-

sive operation, at best, and the authors have pointed out how, by careful study of certain factors involved, material savings can be made. The paper is very interesting and the authors are to be congratulated for the thorough manner in which they have covered the subject.

Referring to Fig. 13, showing the evolution of ring gear containers, it has been our experience that the chimney type container is not practical for direct or box quenching. The chimney interferes with the removal of the hot gears from the container. Containers have been made with the chimneys removable, but oftentimes the chimneys become wedged and their removal causes mutilation of the gears or chimney or both.

Written Discussion: By E. F. Davis, metallurgist, Warner Gear Division, Borg-Warner Corp., Muncie, Indiana.

The authors have given a very excellent treatise on the troublesome box problem and the many factors which affect their life in service. One of the important items causing box and tray failure which the authors do not seem to have discussed is the permanent growth either due to carburizing, graphitizing or coarsening of the grain structure.

It is a well known fact that round boxes or any boxes of tubular construction give the most satisfactory life, and the long rectangular type the least. Corrugation seems to help the latter, but is no assurance against early failure. The writer believes the percentage of iron is the principal factor for ferrite will dissolve carbon and we thus have a chemical reaction within the internal structure of the metal. The gradual increase in length or growth of carburizing trays used in continuous gas carburizing furnaces fully exemplifies this. In one plant some of these grew ½ inch in length after six months' service. Another alloy higher in chromium and nickel did not show this tendency after eight months' service. The boxes higher in alloy content do not fail as readily as those lower in alloy content. The round box will increase in diameter and when the strains become severe will usually flare and crack at the top, but this type of box seems to be the best for longevity and, no doubt, would operate more effectively with lower alloy content than the rectangular box. Heat resisting alloy which has been worked, such as by rolling, is generally superior to the cast metal of the same section and does not seem to be affected as quickly by permanent growth. This can be noted in the difference in life between thin-walled boxes of cast alloy and the rolled alloy, the life being greater in the latter.

I question very much the decrease in heating time between a thin-walled or thick-walled box. The carburizing compound is the poor heat conductor, whereas the metal is a good heat conductor, and although the heat radiation through the metal might be somewhat slower in the thick-walled box, yet the heat must penetrate a mass of carbon grains of high thermal resistance regardless of whether the box walls are thick or thin. The chief economy of thin-walled boxes is in their lighter weight. However, the chimney boxes or anything which increases the surface to heat conduction must aid in reducing the time to bring the boxes to carburizing temperature.

Written Discussion: By C. E. Malley, sales manager, Ohio Steel Foundry Co., Springfield, Ohio.

Mr. Roush and Mr. Dames have presented a very interesting and intelli-

gent study of this subject and have developed a number of important factors influencing container life not heretofore considered in their proper light.

The alloy producers are aware of the varied operating conditions to which containers are exposed and box life (containers from same pattern and of same compositions) may vary fifty to one hundred per cent between two plants.

The failures of conventional type containers to a great extent are caused by thermal shock rather than by inherent casting defects. Due to operating conditions it is sometimes impossible to maintain uniform heating and cooling and the answer seems to be in designs that compensate for this condition.

The special containers for individual parts as described in this paper will no doubt gain considerable favor and represent another forward step in carburizing development.

Depending on the parts to be treated and the furnace facilities, there is a definite application for both sheet and cast containers. The combination sheet and cast containers are steadily gaining favor due to the fact that the advantages of the two can be incorporated in one unit, the advantage of the sheet being the lighter weight and the rigidity and additional strength of the cast parts. The past several years have developed a considerable increase in alloy container life and continued co-operation between user and producer will be of mutual benefit.

Written Discussion: By J. W. Henry, superintendent, Electro Alloys Co., Elyria, Ohio.

Since becoming a member of the Society in 1920 at Cleveland, I have read with much interest the papers sponsored by the American Society for Metals on practical metallurgy. Such a paper, "A Study of Commercial Carburizing Containers," by Mr. Roush and Mr. Dames of the Timken-Detroit Axle Company, is vitally practical and should prove of real benefit to all practitioners of the carburizing art.

A carburizing box to an alloy foundryman is just another casting and yet the evolution of that box into the modern streamlined muffle of today, with its butt welded joints, represents forward strides in the use of nickel-chromium alloys for carburizing.

The composition and physical properties of these alloys have been well covered by the authors and it is on container failures that I wish to comment.

Shrinkage, or the change in volume from liquid to solid, is undoubtedly the most important factor in casting failure. Contraction, or the change in dimensions in the solid while cooling, likewise contributes to failure at times.

Shrinkage, when manifest to the eye as a crack in a newly poured casting, such as a box, is practically impossible to repair because the areas adjacent are badly torn. When shrinkage manifests itself internally only, then this particular area has less density and acts as a notch, leading to cracking after a few heatings and coolings. When no shrinkage is present or rather when the shrinkage is well distributed, and this can only be detected by X-ray inspection, such a casting with reasonable use could be expected to deliver good service.

The distribution of shrinkage must be accomplished by experienced temperature control in casting, by judicious gating, risering and chilling and by controlled directional solidification.

To develop the best casting practice, the X-ray affords invaluable assistance and the company with which the writer is associated has for five years faithfully and diligently pursued a policy of inspection and casting improvement by its use.

I can only agree with the authors that the combination of sound castings, intelligent application and use, and scientific design of furnaces makes for better carburizing at least cost.

It is apparent that Mr. Roush and Mr. Dames have given generously of their time and experience in this paper and deserve much credit and thanks for their fine exposition.

Written Discussion: By John F. Wyzalek, chief metallurgist, Hyatt Bearings Division, General Motors Corporation, Harrison, N. J.

The authors present an interesting review of the carburizing container's evolution. In this connection, it may be of interest to mention that during the transition from cast iron and steel to alloy containers, a serious attempt was made to commercialize a process whereby steel castings were coated with either chromium or nickel. This proved impractical due to inability of the steel to properly diffuse the alloy coated in the mold with section less than about one and one-half inches.

Although the authors do amply touch upon design as being important in regard to container life, it is felt that too great a stress cannot be made on this point since it is a fact that frequently improper design, effecting unequal expansion and contraction during repeated heating and cooling, tends to bring about earlier failure due to warpage and cracking than any other factor under normal operating conditions. Leaving this matter more in the hands of the foundry engineer would prove to most of us beneficial.

Written Discussion: By E. H. Stilwill, metallurgical department, Dodge Brothers Corporation, Detroit.

Having read this paper with a great deal of interest, the authors are to be congratulated on the clear and concise manner in which they have presented it. This subject is of vital importance to the production supervisor in regards to the quality and cost of the parts processed.

It has been our experience in the use of thin-walled, cast alloy cylindrical containers using separate cast iron tubes as chimneys for transmission and axle gears and pinions, and thin-walled, cast alloy tubes for shafts. This practice makes for uniform heating in the shortest time, with the least amount of compound for the results desired with very satisfactory container life.

For small parts that can withstand a mild tumbling, one-half of a revolution per minute, we have found that a cast alloy retort furnace using a solid carburizer will give a high quality product and a long retort life at a low fuel and labor cost.

Written Discussion: By L. D. Gable and E. S. Rowland, The Timken Roller Bearing Co., Canton, Ohio.

We wish to commend the authors for a very able discussion upon a subject difficult of generalization. Our own experience has led us to choose a cast container of an alloy similar to No. 9 of Table I. Such a container gives us the best combination of life and cost. Into the question of container economy the factors of spoiled parts when the container cracks, lost furnace time, etc.,

must also be injected. We also agree that the importance of a proper container-work relationship is often wrongly underestimated. In our opinion, the most important factor in the life of cast containers is proper foundry practice. Early failures can be almost invariably traced to faulty foundry technique. We have been fortunate in recent years to observe the beneficial effects produced by a selection of containers on the basis of X-ray inspection.

The authors state that no difference in the carburizing cycle occurred when the containers were used with and without covers. This is undoubtedly true when highly energized compounds are used but the latter procedure is risky with weakly energized compounds designed to avoid high carbon concentrations at the surface. In addition, the container cannot be as completely filled with work when run without a cover, due to the shrinkage resulting from the exposure of the compound while hot.

Written Discussion: By L. A. Lanning, metallurgist, General Motors Corp., Bristol, Conn.

The authors of this paper are to be commended for their suggestions in connection with the design and application of containers suitable for specialized types of production carburizing.

There is without question a wide field for the application of this type of thinking in connection with the economics of carburizing.

Unfortunately, however, there is a vast number of users of carburizing boxes or containers who are unable to take advantage of these possibilities due to a wide variety of parts to be processed with a comparatively small volume of each. In such cases one type container must be made to serve many purposes regardless of its exact suitability. In these cases container life is the most important item to the user. It has been my experience that the heat hour per dollar varies but little in the choice of alloy, but it has been my experience that in the majority of cases the consumer does not realize the complete value of the alloy, i.e., he does not obtain the maximum life which the alloy is capable of giving. The majority of failures appear to be due to casting defects rather than to oxidation of the alloy. These defects may not occur before the expiration of the heat hour guarantee but do occur too frequently before the usefulness of the alloy has been exhausted.

It seems to me the maker of alloys is under a distinct obligation to the user to make a determined effort to better his casting practice to the extent that the user is able to realize the full value of service which the alloy is capable of giving.

Oral Discussion

H. W. McQuaid: In my opinion there has been too much stress given to the economies which can be obtained in continuous gas carburizing, and particularly continuous gas carburizing as compared to the box type of carburizing. The carburizing rate in the box is practically the same as in gas, and by properly designing containers and the furnace it is my opinion that you can carburize practically any article just as rapidly if not a little more rapidly in carburizing compounds than you can with gas.

O. W. McMullan:[23] The authors have presented a very interesting paper.

[23] Metallurgist, Youngstown Sheet and Tube Co., East Chicago, Indiana.

The first commercial requirement of all is, of course, to produce a satisfactory part. After that the next thing is to produce that satisfactory part at as low a cost as possible. The authors have omitted much reference to the cost of the finished part. I wonder if, in their reply, they would not put in something which would include alloy cost and container cost, because the fabrication cost of some of these complicated designs is considerably greater than the more simple design suitable for packing a number of parts in one large container. Relative figures, at least, on some overall costs, putting in the extra cost of container, and perhaps also differences in container life, would be of interest.

Authors' Closure

The authors are very grateful for the many excellent discussions of this paper, and feel assured that greater accomplishments will result from further study of this subject.

An attempt was made to classify and describe the different types of failures with no mention of the most common or most important type. It is only natural to find a difference of opinion on this, but with careful consideration on the part of the users and manufacturers, premature failures can be minimized and normal container life realized.

Mr. McQuaid's mention of the importance of welding of sheet containers cannot be over stressed.

In answer to Mr. Lanning's question in regard to having a large variety and a small quantity of parts to carburize, we can only say that wherever it is possible to carburize a part in a container designed for that part it will mean a saving in time and an improvement in quality. The paper is not an attempt to set up definite specifications for carburizing containers for all uses. Suggestions and descriptions are given which have proven very satisfactory to the authors and should be useful in other applications.

With reference to the results obtained in gas carburizing versus those of pot carburizing, it is true that most of our conclusions have been based upon a comparison of the most modern gas carburizing equipment with old and obsolete pot carburizing equipment. No doubt much better results can be obtained in pot carburizing with well designed modern equipment, but there are good reasons to believe that the maximum in gas carburizing has not been reached. A direct and accurate comparison is impossible with existing equipment.

Mr. Davis has mentioned a very interesting point in regard to the growth of alloy containers after extended use. It is known that permanent growth does occur and that more growth occurs with the lower nickel alloys, but it is not believed that this should be referred to as grain growth. This growth is believed to be due to the increase in volume, caused by the absorption of carbon, certain types of corrosion products and impurities.

In Mr. McMullan's discussion, reference is made to the container cost that may be expected from individual carburizing in containers of relatively complex design, when compared with mass carburizing of the same parts in boxes of conventional design. Reference is also made to the life of all cast containers when compared with combination containers of the same dimension with sheet bodies welded to cast bases and containers of the "full floating"

type where the sheet body is supported by, but loosely attached to, the base.

In determining the container cost for any carburizing operation, two factors must be considered; container life and container efficiency. Although these factors are closely related, they should be studied separately to obtain a true measure of container performance. While container life, quite generally expressed in terms of heat hours by users as well as manufacturers, provides a record of alloy behavior under given operating conditions, it can hardly be considered a measure of container efficiency. It is quite possible to show excellent records of heat hour performance with containers having wall sections of ½ to 1 inch in thickness and of such dimension that 3 to 5 times the compound needed, is used. It has been the experience of the authors that container efficiency and therefore the container cost should be expressed in the terms of the number of pieces carburized per container dollar invested. Container efficiency is largely dependent on container design, for this factor controls the box dimension and the amount of compound used, the metal thickness and analysis, the ratio of container weight to the loaded container, hence the time necessary to produce the desired case depth and the fuel required.

Considering the first reference, in cases where operating conditions permit a change from mass to individual carburizing of parts (Figs. 25 and 26) in containers of the design shown in Fig. 22 or 27 a reduction in cost per piece of 12 to 20 per cent can be expected in furnaces of the oil-fired batch type.

With the design shown in the figure referred to, it has been possible in actual practice to show a 75 per cent reduction in compound used, 28 per cent decrease in carburizing cycle, 30 per cent reduction in pot weight and an increase in pot life of 15 per cent with an increase pot cost of only 20 per cent. This reduction in cost could be extended to 25 or 30 per cent where the furnace equipment was of the gas-fired continuous type.

In the second reference, where comparison is made between the all cast and combination types of containers it has been the experience of the authors with batch type carburizing in oil-fired furnaces, that the latter type provides the greatest economy. The full floating type of container has shown definite advantages over the all cast container since it is possible, in designs of this type to retain the value of the sheet bodies differing in analysis from the heavier cast base which supports the load. An average of approximately 10 per cent increase in life has been shown with containers of this type, but of different dimension. A large part of the economy realized was obtained by the change in the rectangular container, because of the relatively poor showing made by the all cast box due to early failure of the long supported sides.

Mr. Henry brought out one of the main points we had hoped to emphasize when he said, "A carburizing box to an alloy foundryman is just another casting. . . ."

It is this idea, or the lack of knowledge and appreciation of the problems of others, that has been responsible for the neglect of proper design and manufacture of carburizing containers.

Certainly, if alloy manufacturers, furnace builders, and users of container equipment will acquaint themselves with each others problems, their combined effort will result in more efficient, more economical and higher quality carburizing.

THE PHYSICAL AND CHEMICAL CHARACTERISTICS OF CARBURIZING COMPOUNDS AND THEIR HANDLING IN PRODUCTION

By S. L. Widrig

Abstract

This paper discusses the various types of solid carburizing compounds and the materials of which they are composed. The compounds consist of three principal constituents, namely; charcoal, binder and energizer. In a good compound each constituent is carefully selected to serve their specific purpose in the most economical and efficient manner.

The importance of good adhesion of the energizing agent to the base charcoal is particularly stressed. The proper proportion of energizer to charcoal must be maintained if good results are to be obtained.

It is pointed out that poorly designed conveying systems and excess screening or dusting result in high energizer loss. Energizer lost in handling must be replaced by additions of new compound in order to maintain a proper balance.

Parts to be box quenched are most economically carburized in certain types of compound which are slow burning when exposed to air at elevated temperatures.

THE purpose of this paper is to review the commercial application of solid carburizing compounds. The discussion will be confined to the selection and application of compounds. No attempt will be made to cover the theoretical aspects of the subject or the problem involved in the design of containers or other equipment.

Carburizing with solid compounds is an old art and the materials used have become quite standardized. There are two principal types of compounds on the market at present, either of which when made of the best materials, will produce satisfactory results. All commercial compounds consist of charcoal, an energizer and a

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A paper presented as part of the Symposium on Carburizing presented at the Nineteenth Annual Convention of the American Society for Metals held in Atlantic City, October 18 to 22, 1937. The author, S. L. Widrig, is metallurgist for the Spicer Manufacturing Corp., Toledo, Ohio.

binder. In addition to these three components, various forms of carbonaceous materials, such as coke, coal tar products and anthracite coal, are some times used. The difference which distinguishes the two types lies in the method by which the energizer is added. One type has been made for many years, and consists of a charcoal core to the surface of which a carbonate energizer has been made to adhere. In the other type a more or less uniform mixture is obtained by mixing the pulverized charcoal and energizer with the binder.

The charcoal which is the basis for practically all compounds is usually obtained from hard woods which are selected to give as dense a product as possible. Charcoals for carburizing have also been obtained from various materials, such as coconut shells, peach pits, etc., and producers of compounds using these materials claim that they improve results. It seems that the same properties which are important for gas mask use are also important in charcoal used for carburizing. The density is an important factor in the rate of burning of the charcoal when exposed to air. Since most compounds are exposed to air at elevated temperatures, the burning of the compound becomes an important factor. For this reason the use of soft wood charcoals which burn more readily than the hardwood charcoals are not considered satisfactory. The main function of the charcoal is to insure a continuous supply of carbon monoxide to the work being carburized and to remove from the work the carbon dioxide which results from the breaking down of the carbon monoxide in contact with the work. This action requires that the charcoal be in close enough contact with the work to insure the removal of the carbon dioxide. Since carbon monoxide and carbon dioxide are gases, the charcoal particles should be of such size as to permit free circulation. Too fine a charcoal, or segregation of dust from the energizer, will, in contact with the work, result in a high concentration of carbon dioxide and carburizing will be retarded if not entirely stopped. If too large a particle size of charcoal is used it may be found that in recesses the intimate contact of charcoal and work is lacking, with the result that too high a concentration of carbon dioxide is also present on the work, causing retarded carburizing. Ordinarily, the charcoal size specified is about three mesh and seldom larger than two mesh.

At the present time, barium carbonate is the principal energizer used in commercial compounds. Carbonates have been used in connection with charcoal for a hundred years or more in carburizing compounds. The usual explanation of the function of the carbon-

ates is that they serve as the initial source of supply of carbon dioxide which reacts with the heated charcoal to form the carbon monoxide necessary for the carburizing reaction. It is necessary that a sufficient amount of carbon dioxide be applied to give, after reacting with the charcoal, a continuous supply of carbon monoxide for carburizing. The controlling factors in the selection of a specific carbonate are the temperature and time of carburizing. The less stable carbonates, such as calcium and sodium carbonate, evolve their carbon dioxide at lower temperatures as compared to the more stable carbonates such as barium carbonate.

Most of the carbon dioxide resulting from the breakdown of the calcium and sodium compounds is evolved in a very short time after reaching the carburizing temperature.

For high temperature carburizing and deep cases, a stable carbonate is required. In ordinary practice, calcium, sodium and barium carbonates are used in order to take advantage of the carburizing which occurs on heating through the lower temperature range. As sodium carbonate has a tendency to attack the alloy containers, the percentage used is usually low. The rate of carburization depends upon the percentage of energizer which is in the compound. As the percentage of energizer increases up to approximately eight per cent, there is a proportionate increase in the rate of carburization, but above this percentage there is little, if any, increase in rate of carburization with increasing percentage of energizers.

During the carburizing operation there is a loss in carbonate energizer due to handling, etc. If this loss results in a carbonate content of less than 8 per cent it becomes necessary to add more energizer, which is usually in the form of new compound. If the energizer could be so bound to the charcoal that none would be lost in the handling, the amount of new compound required would be small indeed.

The heat resisting alloy carburizing box of today with its relatively tight fitting cover does not permit much burning of the compound during the carburizing operation. The shrinkage because of burning with a properly designed box occurs for the greatest part in the quenching of direct quenched work.

If covers are a poor fit, there will be some loss in the carburizing operation, not as a rule, however, exceeding 8 to 10 per cent on mixtures containing the minimum percentage of new compound. The largest single loss which requires a high percentage of new com-

pound is that from rough handling. This rough handling results in loss of energizer in the form of dust. The use of high speed conveying systems for handling compound or excessive screening is certainly to be condemned. Screening to remove dust is a common operation in most plants and is usually carried to extremes. Screening of compound to remove dust, clay, scale, etc., should be the very minimum to insure freedom from excessive dust which might lodge in the holes, narrow slots, etc., and cause a thin case. Care and thought given to reduce dusting will be well repaid since most of the dust is energizer and its removal is costly. The depletion of the energizer by dusting can be reduced either by the use of a more adherent binder or by more careful handling or by both.

Every per cent of energizer which can be retained in the used compound reduces the new material required by a very large amount, depending on the total retained percentage. The theoretical addition of new compound is largely governed by the loss in energizer in the used compound and by the burning and should be calculated to give a minimum of 8 to 10 per cent of energizer in the mixture.

While it is necessary to add only a sufficient amount of new compound to replace the depleted energizer, it is customary to add new compound to the old in a fixed percentage. Many plants are using a percentage of new compound far in excess of that required. Since the loss of energizer depends upon the care used in handling the compound, there will be a variation in the amount of new compound required from one plant to another depending upon the methods used as well as the type of compound. With reasonable care under ordinary conditions, not more than 20 per cent of new compound should be required, and if an attempt is made to reduce the loss in energizer the percentage can in most cases be carried as low as 10 to 12 per cent. The carbonate energizers are usually used in a powdered condition. They are used in this condition because it facilitates intimate contact and adherence to the charcoal base. It is not, however, necessary that the energizer be used in the powdered form or that it adhere closely to the charcoal base. It is extremely important, however, that the energizer be of such size and so distributed that the carbon dioxide formed by its decomposition on heating will react with the charcoal.

At one time a carburizing compound was quite extensively used in which the energizer was placed on the bottom of the carburizing box and the work in the charcoal above the energizer. While this

compound worked quite satisfactorily under ordinary conditions, the handling problems made it undesirable. Powdered energizers dusted onto the charcoal will, when properly handled, produce satisfactory results but due to the loss of energizer in handling they are not economical. The use of water in which carbonates are suspended as a means of introducing the fine powdered carbonates into the pores of the charcoal has also been widely used with satisfactory results. It requires, of course, careful drying and is subjected to the criticism that the bond between the energizer and the charcoal is weak and hence the loss of energizer in use is high.

Since the performance and economy of a given compound depend so greatly upon the retention of the energizer on the charcoal, we find that the method of maintaining the energizer in contact with the charcoal becomes of the greatest importance. A careful study will reveal that the use of expensive binding material is usually warranted as compared to the cheaper but less permanent method of maintaining adherence of compound and energizer.

In the past there have been several common types of binders used in maintaining this adherence of energizer to charcoal. They can be roughly classified as "dusted on," water binder, oil binder, molasses and other organic compound binders, and tar binders.

The relative efficiency of these materials as binders is roughly in the above order. The use of a tar binder, which is coked before use, when properly made is probably the most satisfactory of all binding material, with the molasses binder a close second. It is the writer's opinion that the use of "dusted on" energizer, or water bound compound, is only commercial in those plants which deliberately discard used compound in amounts which greatly exceed the necessary minimum without regard to possible economy which can be obtained by using more efficient binders. The selection of a compound should be based upon a very careful study of the conditions which exist in a given plant, and, if necessary, the equipment should be changed to permit the use of a more efficient compound. It is safe to say that properly made compound with a molasses or tar binder can always be made to produce lower cost and more reliable carburizing than the "dusted on" or water, or oil compounds.

If coated charcoal energizer combinations are used when material is quenched directly from the box, the coked tar binder has some advantage, in that the coating of coked tar protects the compound from burning and improved economy is to be obtained. With

the great increase in the amount of work which is quenched direct from the carburizing box and which has taken place in the last five years, the burning of the compound has become an important item. This procedure has also increased the desirability of the "pill type" of compound in which there is a uniform mixture of pulverized charcoal energizer and binder. This compound, while considerably more expensive than the water or oil bound type, is found in many cases to be economical where very much direct quenching is the rule, and is not subject to loss of energizer which occurs with the usual coated type and maintains its efficiency with a smaller addition of new material.

In the selection of a compound the initial cost should be of secondary importance as compared to the cost of the compound per pound of work produced and this depends to a large extent upon the new material required in the mixture. If careful study is given to the determinations of the correct percentage of new compound required for maximum carburizing efficiency, it will be found that some of the most expensive compounds will produce work at a lower cost and with less variations than with the cheaper types of compound.

The use of diluents such as coke or other inert substances in compounds are not as common as in the past, but most of the coated compounds contain at least 20 per cent of coke which serves to reduce the cost of the compound, to decrease the shrinkage and to decrease the insulation effect of the compound. Possibly, the most important single reason why compound carburizing has been considered slow, in comparison to gas carburizing, has been the increase in carburizing time caused by the insulation effect of the compound itself. The coke used reduces the time lost in transferring of heat and results in decreased time required to bring the work to temperature. The usual tendency to use a large ratio of compound to work results in an unnecessarily long carburizing cycle due to the slowness with which the work comes to temperature. The insulation effect of the compound is of great importance and only the minimum amount of compound should be used which will result in satisfactory carburizing. This in most cases is very much less than the amount which text books would indicate as correct. It has been the writer's experience that, with the development of modern carburizing boxes, a greater increased percentage of work to compound can be used than was usually thought possible. This is a subject to which every operator of a carburizing department can give consider-

able study in the endeavor to arrive at the most economical ratio of work to compound and to determine the very minimum of compound which could be used in producing the desired results. Any increase in the percentage of compound above this minimum will result in increased time and carburizing cost, because of the insulation effect of the compound.

It is recommended that a very maximum amount of work which can be introduced into a given container be used with only a thin layer of compound to separate the work. It is impossible in a paper such as this to give specific requirements as to the arrangement of the work in a carburizing box or the amount of compound which should be used. It is believed that in practically every plant in the country a much larger percentage of carburizing compound is used than that which is necessary to produce the maximum results and it is hoped that this subject will be given consideration by those readers of this paper who are reponsible for the practical operating economy of a commercial carburizing department.

DISCUSSION

Written Discussion: By H. J. Bates, metallurgist, Fairfield Manufacturing Company, Lafayette, Indiana.

The author, S. L. Widrig, is to be congratulated upon this practical discussion of a material which is quite common in the commercial heat treating field, but which has been infrequently discussed in the Society's technical literature during the last few years.

On the sixth page of Mr. Widrig's paper reference is made to the relation of initial cost of compound compared to the cost of compound per pound of work carburized. Not only does this cost depend upon the new material required in the mixture and the other factors which were mentioned but consideration should also be given to the space occupied by a unit weight of the compound. The compound is purchased by the ton but used on a volume basis. The table below shows the volume occupied by a ton of five different carburizing compounds.

Compound	Volume (Cu.Ft.) Occupied by One Ton of New Compound	Approximate Per Cent Change (Shrinkage) After One Run of New Compound	Type Compound
A	59	slight	"charcoal core"
B	82	15	"charcoal core"
C	85	12	"charcoal core"
D	102	20	"charcoal core"
E	51	11	"pill"

It will be noticed that there is a 100 per cent variation in the space occupied by a unit weight of the new compound.

Written Discussion: By L. E. Webb, metallurgist, Frost Gear & Forge, Division of Clark Equipment Co., Jackson, Mich.

I want to congratulate the author, Mr. Widrig, on his excellent paper in which he has so ably covered the subject of carburizing compounds. The information contained in this paper is of such value practically and theoretically that the ordinary heat treater, or layman, can readily understand it.

It may be of some interest to describe a carburizing compound now in use at our plant. The base materials of this compound are properly sized pure coal tar carbon and corn cob charcoal. The coal tar carbon being one of the purest forms of commercial carbon having a fixed carbon content of approximately 95 to 98 per cent, with an ash content of approximately 1 per cent.

In its manufacture the base carbons are energized with the usual activators. These activated particles are then coated with a shell of pure carbon and then calcined at steel treating temperatures. This shell or coating prevents the loss of the energizing material and also adds considerably to the mechanical strength of each pellet or particle. It is, therefore, apparent that the dusting loss is very low, due to the wear resistant properties of the carbon shell together with the fact that the energizing material is sealed within.

There are always certain advantages and disadvantages to be found in every compound. Some of the advantages to be gained through the use of this compound might be listed as follows:

(1) Low shrinkage.
(2) Low mechanical loss—Due to the mechanical strength of the pellets or particles, the material will withstand fairly rough handling.
(3) Cleanliness—The fact that the dusting percentage is low, makes working conditions much better.
(4) Economy—The per cent of addition is much lower, thus obtaining more compound life and more weight of steel carburized per pound of compound.
(5) Nonburning quality—This allows the quenching of material direct from the box.

It is, of course, only fair that some of the disadvantages be noted such as:

(1) Slight increase in weight per cubic foot.
(2) Increased insulating effect.
(3) Time required to reach carburizing temperature may or may not be slightly increased.
(4) Volume build-up due to low shrinkage.

It has been our experience that the activity of such a compound as described does decrease fairly rapidly at temperatures below 1650 degrees Fahr. whereas charcoal base compounds usually retain their activity at somewhat lower temperatures. The actual physical strength of a material, especially when heated, is important, in that it determines the supporting power of the material and the liability of parts to distort when packed in it.

In materials containing an energizer, the life depends a great deal upon the ability of the material to regenerate. Sufficient aeration is, therefore, of importance and should be made a standard routine in handling carburizing materials.

Surface decarburization usually occurs only with a weak carburizer and at low carburizing temperatures; however, surface decarburization never occurs when a reducing atmosphere is maintained by the presence of hydrocarbon gases.

Carburizing materials should be kept dry as moisture leads to pitted work and soft spots.

The author states that the tendency is to use a large ratio of compound to work which is quite true especially so in shops having a large variety of parts. This may be partly due to the standardization of pot size. I believe it is quite common to find that a ratio of two parts of work to one of compound is being used and I agree heartily that we should make every effort to decrease this ratio.

Written Discussion: By Hugh Rodman, president, Rodman Chemical Co., Verona, Pa.

Mr. Widrig's paper contains a number of valuable suggestions. Probably the two most important are those regarding the handling of carburizer and the careful selection of carburizing pots.

There is no doubt that carburizing compound is injured by violent screening. Not only is the energizer shaken loose from the surface of smeared compound but the carbon structure itself is frequently abraded with loss both of energizer and carbon. Where coke as well as charcoal is employed in smeared compounds, the troublesome accumulation of coke is increased since the coke abrades less easily than the charcoal. Even where strong binding agents, such as molasses and tar, are employed to bind the energizer upon the surface of the carbon fragments, severe loss of energizer may occur since the structure of the charred binder is frequently weak.

The careful selection of pots to fit the work merits consideration. Unquestionably, as suggested by Mr. Widrig, a considerable saving in furnace time and amount of carburizer could be secured by more careful selection of pots. In addition, greater uniformity of work might be expected.

Mr. Widrig's paper disclaims any consideration of theory but he has made several observations which should be corrected. At least one commercial carburizer contains no charcoal. Finer sizes of uniformly mixed carburizer will carburize more rapidly than coarser sizes once they have been heated throughout. The trouble with these finer sizes is low thermal conductivity. The carbonate energizers serve to produce catalysts as well as to supply carbon dioxide. These are minor criticisms of an otherwise very excellent paper for which the author should be complimented.

Written Discussion: By L. L. Ferrall, metallurgist, The Timken Roller Bearing Co., Steel and Tube Division, Canton, Ohio.

Mr. Widrig has presented a very thorough description of the make-up, properties and methods of handling of carburizing compounds as they exist in present day carburizing operations. It is difficult to add to the paper in any other way than to mention some practical experiences.

It seems that the real problem in carburizing compounds is to select that compound which will economically produce the type of work desired. Early experience in carburizing large bearing parts requiring deep cases in which carbon concentrations were quite important definitely showed that the car-

burizing compound had to be designed for the job. A compound which showed considerable shrinkage introduced difficulties, likewise compounds which were too fast or too slow in carburizing gave undesirable structural conditions in the cases. Compounds were formulated in co-operation with the manufacturer which produced the required types of cases. Having once arrived at the proper combination it was possible to write specifications which would insure duplication of the compound.

The specifications require that the compound must have a constant weight to volume ratio (this controls the type of charcoal used), that it must not show more than a definite percentage of shrinkage when subject to a definite time-temperature cycle, and that it must produce, under a definite carburizing cycle with given test pieces, cases of definite carbon concentration. The carbon concentrations are determined by analyzing successive carbon cuts. These tests insure proper amounts and proper introduction of the energizer.

The matter of charcoal or particle size is often discussed and no doubt can vary to some extent in carburizing different parts. Our experience has indicated that a 4 to 5 mesh is quite satisfactory for general usage. The amount of compound around the work should be determined with consideration of the particle size of the compound. A coarse compound really should require a thicker layer since along with it we may expect large voids which in turn reduce the CO concentration. On the other hand a thin layer of coarse mesh compound will tend to let the load concentrate and this may even damage the work by marking or distorting. It has been found quite important to surround the work with a uniform layer of compound in order to promote equal heating, which in turn is reflected in uniform cases and consequently uniform performance in the subsequent hardening operations.

Written Discussion: By C. F. Lewis, Cook Heat Treating Corp., Houston, Texas.

The author has so thoroughly and competently covered this subject of carburizing compounds that there is very little that can be added. The writer, however, has had the privilege to be quite closely associated with the use of carburizing material, utilizing, as the author mentions, peach pits as a source of charcoal; and since, due to geographical location and transportation costs, this type of compound is marketed principally on the Pacific Coast. A word or two concerning its characteristics may be of interest to many compound users in the East.

In a commercial heat treating shop the cost of carburizing compound is a very important item of expense. If the enterprise is to show a profit on this operation, advantage must be taken of anything that will lessen the cost per pound of steel carburized. Losses occur, as pointed out by the author, through screening and through burning of the charcoal when exposed to the air. Screenings, of course, contain both energizer and pulverized charcoal.

In the great majority of commercial shops, the compound is handled manually with a shovel, with its attendant tendency to crush and pulverize the compound. A charcoal, then, must be selected that is hard enough to resist this action, and it is the writer's experience that peach pit charcoal does this to a greater extent than even the hardwood charcoals. We have found in the writer's plant, however, that the screen losses are negligible when com-

pared to losses from spillage and careless handling on the part of the operator.

Regarding the loss of energizer, due to the heavily serrated surface of a peach pit and its inherent porosity, most of the energizer is contained within the charcoal particle itself and does not come in contact with the handling equipment. On a smooth surfaced char, even with the best of binders, energizer is lost mainly through the rubbing action of one particle against another, and where these smooth surfaces are absent, loss of energizer from this cause is greatly reduced. It is not uncommon in the writer's shop to mix only one part of new material with 10-12 parts of old in order to maintain a satisfactory concentration of energizer.

Losses due to burning of the hot compound when quenching direct from the pot are reduced in many commercial shops by spraying the surface of the material with water, and this brings up the question of moisture, which the author has not mentioned. It is the opinion of many metallurgists that hygroscopic moisture in the compound is a source of soft spots, and some even go so far as to prohibit the use of carburizing compound that has been shipped by water freight, because of the possibility of moisture absorption. It is hard to conceive that small amounts of water heated to some 1700 degrees Fahr. in the presence of carbon can remain as steam and do harm to the work. As a matter of fact, water gas is made in just this manner according to the following reaction:

$$C + H_2O \rightarrow CO + H_2$$

Neither of these resulting gases can harm the work being carburized—the H_2 burning as it escapes to the furnace or being absorbed by the charcoal; the CO being utilized by the carburizing reaction. The writer has never been able to definitely trace soft spots to this source, and it is his opinion that entirely too much weight is given to this subject of absorbed moisture in carburizing compound.

One point that should be brought out as a characteristic of peach pit compound is that, after extensive investigation, the War Department has found that a charcoal made from peach pits will absorb more gas per unit of weight than charcoal made from any other material, and hence has adopted it for use in gas masks. This property becomes useful during the early stages of the carburizing cycle where certain inert gases, such as nitrogen, must be absorbed.

The author points out the fact that barium carbonate is to be preferred to the sodium compounds for the energizer. This concurs with the writer's experience, not only for the reasons cited, but also for an entirely different one that recently came to his attention. Where parts were partially packed in fireclay or sand to prevent local carburization, a sodium energized compound was found to form a hard glass-like substance on the surface of the work that is extremely hard to remove. This substance did not occur as a thin film but as small spherical globules scattered at random over the surface. Not only was it objectionable from the standpoint of grinding wheel wear, but when chipped off left small pits that sometimes prevented the part from cleaning up. A change to a barium energizer eliminated this trouble completely and led to the thought that this substance is actually a fusion product

very similar to certain types of glass. In addition to the above, it is a definite loss of the all-important energizer.

The author mentions particle size and its effect on the case. It is the writer's opinion that satisfactory cases can be obtained by using much larger sized compound than is commonly marketed by most manufacturers. This is substantiated by the fact that the writer has almost doubled the particle size of his compound without noticeable effect on the quality of the case. It should be interesting to conduct a series of experiments to determine, for a given set-up, the maximum particle size that can be used, bearing in mind that one buys compound by weight and uses it on a volume basis. If by increasing the size of the compound, one may reduce the weight required to fill a given volume, a definite lowering of the net cost per unit of volume used may be effected.

The matter of insulation effect of the coated compound and its resultant increase in time for a given case is quite important. The use of a diluent in the form of coke does, as the author points out, materially help. The writer has seen experiments run with a compound using up to approximately sixty per cent of coke, and so far as he was able to judge without laboratory analysis, the case was still quite satisfactory for many applications. It must be mentioned, however, that the particle size had to be reduced somewhat in order to give better distribution of the energized compound.

Metallurgists are well aware of the fact that the problems confronting the commercial treater are entirely different from those of the production line. In the one case, thousands of identical pieces are run through the furnaces almost continuously, and the process lends itself readily to very fine adjustment of the several variables. In a commercial shop, on the other hand, not only are there parts of every conceivable size and shape, but also two or three different types of steel may be represented, all in the same heat and often in the same box. He then must strike an average set of conditions, and do the best he can to earn his company a fair return.

Both classes are greatly benefited through improvement brought about by such timely and thoughtful papers as Mr. Widrig's. He is to be congratulated on this contribution to a fine old art.

Written Discussion: By H. E. Martin, E. F. Houghton & Co., Detroit, Mich.

The author has given an excellent review of the composition and utilization of carburizers. It occurs to me that of the vast number of experiments conducted in every plant where carburizing is done on a large scale there is a small portion of this work published and it is a paper such as this that should stimulate more expressions of opinion.

Considerable improvement in methods of handling carburizers in production has been accomplished due to the increase of box quenched carburizing. On one large installation, equipment has been added to cool the compound after dumping from the boxes. After cooling it is conveyed pneumatically to a dust collector where the fines are removed and compound of the proper mesh falling into a used material hopper. By means of a screw conveyor at the bottom of this hopper and in a companion hopper carrying the new compound the

speed of the screws will determine the mixture of old and new compound to be delivered to the loading deck.

Another large carburizer cleaning installation incorporates a cooling chamber but the conveying and screening devices are all mechanical in contrast to the pneumatic system.

Both of these installations were designed to remove fines with a minimum of abrasion on the compound.

Written Discussion: By W. P. Eddy, Jr., Yellow Truck and Coach Mfg. Co., Pontiac, Mich.

I would like to add, to Mr. Widrig's excellent summary of the characteristics of carburizing compounds, some specific information concerning a coked tar-bound material. This compound contains only 15 per cent charcoal, and includes as energizers 15 per cent barium carbonate, 3 per cent calcium carbonate and 2 per cent sodium carbonate. The compound is appreciably heavier than compounds consisting mostly of charcoal, and, since carburizing compounds are ordinarily purchased in weight units and are used in volume units, this is an economic disadvantage of the high coke compound. However, the material is purchased to a stipulated maximum density of 30 pounds per cubic foot, and rebates obtained for excessive weight. (Actually, this compound, as received, usually weighs approximately 33 pounds per cubic foot.)

However, the combination of the high coke content, the initially high energizer content and the excellence of the binder results in a fast heating compound, which suffers low shrinkage, burning and dust losses, and which can therefore be used with unusually small additions of new material. Following are typical results of analysis for carbon of successive 0.005-inch cuts turned from 1-inch round test bars after carburizing. The bars were carburized under production conditions, being packed with other work in cylindrical boxes 16 inches in diameter and 13 inches deep, placed in a batch-type furnace at 1650 to 1670 degrees Fahr., heated for a total time of 13 hours, direct-quenched in oil, and tempered at 1200 degrees Fahr.

Carburizing compound mix	Bar No. 1 9% New Per Cent	Bar No. 2 7% New Per Cent	Bar No. 3 All Old Per Cent
Carbon, first 0.005 inch	1.08	1.08	1.05
Carbon, second 0.005 inch	1.03	1.00	1.03
Carbon, third 0.005 inch	0.95	0.91	0.94
Carbon, fourth 0.005 inch	0.89	0.81	0.82
Carbon, fifth 0.005 inch	0.77	0.74	0.69
Carbon, sixth 0.005 inch	0.67	0.59	0.57
Carbon, seventh 0.005 inch	0.58	0.52	0.49

The total case depth, as nearly as could be measured, was 0.060 inch on each of the three bars. It will be noted that no significant difference among the three samples exists in the outer 0.015 inch of the cases. In production, this compound has been used by mixing 1 part (9 per cent) new with 10 parts (91 per cent) old material; total losses are sufficiently low so that no diminution of the supply of ready-to-use compound occurs.

Written Discussion: By H. B. Knowlton, metallurgical engineer, International Harvester Co., Chicago.

Mr. Widrig has prepared a very excellent review on the subject of car-

burizing compounds and their handling in production. In the main, we agree with the opinions which he expresses.

We are very glad to find that he calls attention to the evils of rough handling of carburizing compounds, and the fact that the efficiency can be decreased by loss of chemical energizers. We do not believe that this point can be stressed too much.

We would like to go a little further than Mr. Widrig has done, and state that rough handling not only causes a loss of chemical but it causes loss of volume of compound, due to the fact that charcoal and to some extent other materials used in carburizing compounds, are friable and easily broken down to dust.

Several years ago the writer was working in a plant where the common practice was to use two parts of old and one part new compound. A series of tests were run in which the compound in an individual box was weighed before and after carburizing, and again after screening. It was found that the total loss of volume could be reduced to as low as 2 per cent by careful handling and still maintain carburizing efficiency.

However, when it was attempted to run the carburizing department on a basis of four old and one new (an addition of 20 per cent of new), we ran out of old compound showing that the loss of volume due to rough handling was in excess of 20 per cent. A better handling system was designed and built, and was operated successfully on the basis of additional new material of 10 or 12 per cent, or about the same figure as given by Mr. Widrig.

With regard to screening, however, we are very much in favor of thorough screening to remove all fine particles whether chemical or charcoal dust. In this connection, it must be remembered that the chemical energizers themselves produce decarburization if in direct contact with the steel. This can be easily demonstrated by placing a layer of chemical in the bottom of a carburizing container with charcoal or a carburizing compound above it, and placing a test pin vertically in the container so that it is in contact with both the chemical and the charcoal. It will be found that the part in the chemical will have little or no case.

We believe that whenever there is sufficient loose chemical in the carburizing compound, it is possible for the steel to come in contact with a small volume of pure chemical, and under this condition there will be danger of soft spots in carburizing.

With regard to the amount of chemical necessary to keep up carburizing activity, we do not entirely agree with Mr. Widrig's statement of 8 per cent, although this is a safe recommendation for commercial practice.

Some years ago we reported a series of experiments, which were run in duplicate and run as accurately as we knew how, to determine the effect of varying amounts of chemical on carburizing activity.[24] Materials containing from zero to 28 per cent of chemical were tried.

By plotting depth of total case, hypereutectoid plus eutectoid zone, hypereutectoid zone against the per cent of chemical, it was found that the effect of the first 2 per cent of chemical was enormous, while 8 per cent of chemical

[24] "Economical Re-use of Solid Carburizing Materials," TRANSACTIONS, American Society for Steel Treating, Vol. XVI, October 1929.

was only slightly better than 2 per cent. In fact, all of the curves were fairly flat from 4 per cent of chemical on. The results may be briefly summarized as follows:

Chemical Per Cent	Hypereutectoid	Hypereutectoid Plus Eutectoid	Total Case
0	0.000	0.020	0.038
2	0.015	0.030	0.050
4	0.015	0.032	0.051
8	0.018	0.034	0.053
20	0.018	0.035	0.052

These tests were made using loose mixtures of chemical and charcoal. However, they were mixed very thoroughly so as to prevent segregations of chemical. With higher percentages of chemical, we found somewhat less uniform carburizing.

We are bringing out this point particularly as we believe that the dangers of loss of chemical due to screening are less serious than the dangers of presence of much free chemical due to lack of sufficient screening. We agree with Mr. Widrig that the screening should be just enough to remove the dust, but would rather err on the side of overscreening than underscreening.

With regard to his discussion of types of compounds, we found the usual statement that sodium carbonate attacks carburizing containers. This seems to be generally accepted, although we have not seen any actual data on this subject. We would like to see this more thoroughly studied.

One advantage of sodium carbonate is that it is water soluble, consequently, it is possible to deposit this chemical all through the pores of charcoal by soaking the charcoal in a sodium carbonate solution. This must be followed by thorough drying. Such a compound should not yield much free chemical. Whether such a compound if kept clean would attack boxes should be actually determined.

We agree with Mr. Widrig's opinion that the use of water as a binder for insoluble chemicals is very inefficient and is not recommended.

Written Discussion: By E. E. Alexander, Caterpillar Tractor Co., Peoria, Ill.

It would have been an interesting addition to the paper to know just what set-up the author had for checking the loss of energizer after each carburizing heat, and if the additions of new carburizing materials are based on actual tests made regularly or if the additions were made at a fixed percentage to replace shrinkage rather than loss of energizers.

Our experience with carburizing materials that have energizers dusted on, water or oil treated, have never been satisfactory in that we could not produce uniform case depths or consistent carbon penetration. We are of the opinion that a material which has been impregnated with the energizers will be free of excessive dust which in most materials is wasted energizers, and in addition materials that have been impregnated with energizers not only have the ability to regenerate but have increased physical strength that helps to prevent waste due to rough handling.

In the use of coke in materials, we would be interested to know if, as the ratio of charcoal to coke changes after constant re-use of the carburizing

material, was it necessary to increase the percentage of energizers rather than just make additions to replace volume losses to maintain uniform results?

We would appreciate if the author could give us from his own experience what he considers a good working ratio of work carburized to material used and whether this ratio would change very much for small and large containers.

We fully agree that there is a general tendency in most plants to use a larger percentage of carburizing compound than is necessary to produce desired results.

Written Discussion: By J. S. Ayling, Case Hardening Service Co., Cleveland.

The paper as presented by S. L. Widrig deals very well with the subject and the following remarks are offered as supplementary. It is evident that Mr. Widrig has discussed only the smeared or coated types and may not be familiar with the impregnated compound which is being widely used by many consumers.

The sodium energized type has several advantages when properly manufactured as by the impregnation process. For example, a smaller percentage of energizer is needed when sodium is used (normally 7 to 9 per cent is employed). Sodium is soluble in water and can be soaked into the pores of the charcoal, producing a very uniform distribution of energizer within each charcoal particle. After such soaking it can be dried and screened and handled without appreciable loss of energizer. Temperatures for carburizing can be used, with approximately the same speed of penetration, which are lower than required by compounds with other types of energizers.

As to the tendency for chemical attack on alloy containers, much of the propaganda regarding this has been disproved. The writer's experience is that because of greater activity of the sodium compound, larger volumes of gas are formed which possibly may have a scouring action on the edge of poorly designed containers. We have yet to see a sound alloy casting which has been in any way chemically attacked by sodium carbonate itself. One of the least destructive neating baths which can be used in alloy pots is composed largely of sodium carbonate.

In discussing charcoals it can be pointed out that there is a vast difference in various grades of hardwood charcoal, due to the kind of raw material, the manner and length of time the wood is aged before charring, and the actual charring process. The preparation of such charcoal is an art in itself. Charcoal made from many types of hard woods, such as birch, beech, maple, etc., is inferior to oak charcoal, especially oak from high ridge lands, which weighs green considerably over 5000 pounds to the cord, as compared to other hard woods weighing around 1000 pounds to the cord. This oak charcoal is extremely slow burning, very hard, and when ground properly makes a chunky square particle as compared to the thinner, long, splintery types often produced from other hard woods. Also it is claimed that charcoal produced in the retort process where the wood chemicals are the main product and charcoal the by-product will be inferior to charcoal produced in kilns where the charcoal is the sole product. It is then worthy of note that the quality of the charcoal-type compound hinges very materially on using the proper charcoal for the base.

The point about screening is well taken by the author. Excess screening with many types of compounds carries off a large proportion of the energizers

unless they can be impregnated into the pores of the base materials, or otherwise securely bound to the particles. In the impregnated type there is not over 1 per cent of sodium energizer on the surface of the charcoal grains. In line with Mr. Widrig's remarks about the use of powdered energizers placed in the bottom of the box with the base material added above, this practice is not now used to any great extent but it recalls experiments conducted by H. B. Knowlton several years ago. Mr. Knowlton used a series of containers, packing several with the energizer in the bottom above which the charcoal was packed. In these, test specimens were placed, one in the energizer itself, one in the charcoal immediately above the energizer, one about the middle of the charcoal, and one at the top. Other containers were packed with various standard commercial compounds. In the first described containers, the sample in the energizer showed no carburization, the one immediately above on the bottom of the charcoal had the deepest case, and the one in the center less, while the one at the top showed considerably lower penetration. The obvious deduction is that for uniform results the energizer must be in intimate contact with the base material. The samples from the compounds were all uniform regardless of position in the container. The commercial compound energized with sodium carbonate gave greater case depth at lower temperatures but at high temperatures there was less difference due, probably, to the greater activity of the energizer at lower heats.

Oral Discussion

R. S. ARCHER:[25] One point which has been raised during this session is that of the relative cost of pack carburizing and gas carburizing. We have had two apparently conflicting statements, both from men who know what they are doing and men who have had a great deal of carburizing experience. It would seem that there has been something lacking in the analysis. That is, perhaps neither of these statements is generally wrong, and perhaps the specific conditions determine which is right. It would seem profitable that we should have some further discussion now.

H. W. MCQUAID:[26] I believe the pack carburizing properly carried out can be just as economically done as in the gas, if not better, but of course that has to be proven or should be demonstrated.

I can think of an automobile company I visited last week where they are carburizing ring gears 0.045 to 0.055 inch in 10 hours. 120 ring gears per hour can be carburized in cylindrical type containers in a conveyor furnace, not of the best design. The conveyors are not the best and they are using more compound than they should. They have studied rather carefully the use of the gas carburizing. In fact, they have at this plant some very extensive gas carburizing equipment which they use on light cases. Gas carburizing is primarily an advantage where you wish to control the carbon in the case, but in the parts where you want to get maximum carbon content, if you will build a box to conform as close to the work as you can get it with a minimum amount of compound between the work and container, approximately a quarter

[25] Metallurgist, Republic Steel Corp., Chicago.
[26] Metallurgist, Republic Steel Corp., Cleveland.

of an inch, and design the furnace with the same care as you do the gas carburizing furnace, so that when the containers come out the men can work around them without getting burned up.

Most of the continuous carburizing furnaces using compound which I have seen have been old furnaces with the containers coming through at all angles, and when they do come out where it is necessary to direct-quench them, the men work under a tremendous disadvantage.

When you design a gas carburizing furnace, the parts should come out to an insulating chamber where the men can reach them comfortably and quench them.

I do not want to start any discussion or controversy, but there is always a tendency when new things are developed to overdo it without giving consideration to what might be done to the old before you rush into the new. The continuous gas carburizing furnace is an ideal method of producing cases of certain types for certain purposes.

R. S. ARCHER: Mr. McQuaid, I wonder if this factor enters into it—that where the nature of carburized work is such that fairly elaborate fixtures are required in the gas process, then the cost of alloy and the necessity of carrying those fixtures may tend to run that cost up, whereas in pack carburizing the parts are easily supported by the compound, whereas on the other hand many parts are carburized in which that is not a problem and no special fixtures are needed.

H. W. MCQUAID: That is particularly true in transmission gears where they carburize entire sets of transmissions at one time and quench them at one time. The fixtures required for those with gas carburizing are expensive and they are difficult to design, but pots to conform to these various type parts are generally designed so you do not get the minimum insulating effect from your compound. In any case the design of a furnace for modern compound carburizing should be the subject of considerable study before we go ahead and put in a $20,000 continuous gas carburizing furnace. We should very carefully investigate what can be done with the right amount of compound and the right fixtures and the right ratio of old and new compound.

I purchased a continuous gas carburizing furnace once, for carburizing gears, and I told the boss when I bought it, "I don't know whether this is going to work but I think I can make it work; if it doesn't I believe we could push boxes of gears through there just about as cheaply as in the container." This furnace today is running on gas, which indicates that there are advantages. They are primarily one of ease of handling. I doubt that the economy obtained is very great as compared to what would be obtained in those same furnaces equipped with proper discharge outlets and proper box design. Those figures have not been developed yet.

One of the best answers to the whole question, as I see it, is the carburizing of ring gears in the automobile plants, where practically all the gears are carburized in compound.

L. D. GABLE:[27] I do not believe Mr. McQuaid or myself can make the general statement that pot carburizing is cheaper than gas carburizing. I think

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[27]Metallurgist, Timken Roller Bearing Co., Canton, Ohio.

that local actual conditions influence the cost on both of those processes. In my own experience, we do both pot and gas carburizing in our plant. I can say that the average cost for gas carburizing in our plant is about one-third of what our pot carburizing is, and I believe I agree with Mr. McQuaid that conditions of containers and compounds and gases used and all those things influence it. Of course, if either process is not efficient, then you are going to have the matter of cost in the favor of the other process.

Author's Closure

The descriptions of the various compounds are very interesting. Undoubtedly the selection of a compound is involved by many factors, such as, cost per unit volume, handling equipment available, whether the work is to be pot quenched or reheated, rate of burning, heat transfer, etc.

The discussion by Mr. Lewis is of unusual interest to many of us who are unfamiliar with peach pit charcoal. With reference to the glass globules, this condition seems to occur quite often where sand is encountered even when the sodium carbonate content is below 2 per cent.

Mr. Knowlton's discussion is both interesting and instructive. From a theoretical standpoint the author agrees with him concerning the percentages of energizer necessary. As suggested, the deleterious effects of sodium carbonate upon alloy containers should be studied and proven. The author is skeptical, however, because in his experience he has had less trouble when the sodium carbonate is held below 2 per cent. Several years ago the author was employed by a firm which used a mixture of sodium and barium chloride for a hardening bath. The use of alloy pots with the particular "set up" was practically prohibitive. The metal deteriorated more rapidly than did either cast iron or cast steel. It was claimed that the failure of the alloy was due mainly to the attack of the nickel by the sodium.

In reply to Mr. Alexander the author has found that commercially it is only necessary to replace the compound lost through screening, handling, burning, etc., by new compound. In other words, assuming a good compound is used, it is only necessary to add sufficient new compound to obtain the original volume. This amount will, of course, vary depending on care of handling, burning, shrinkage, etc. New compound thus added will be sufficient to replace loss of either energizer or charcoal.

Regarding the remarks by Mr. McQuaid, it seems apparent that he is of the opinion that furnace and container design has not been given the proper attention for efficiency in pack carburizing and that if sufficient study be given to the problem, the results of compound carburizing would be comparable if not superior to gas carburizing, particularly in reference to speed of carburization. In this the author heartily agrees with Mr. McQuaid. A series of comparative tests were made at our plant and we believe that where a variety of work is encountered and a variety of case depths are required, pack carburizing can be carried on more efficiently and with as great a speed as gas carburizing.

COMMERCIAL GAS CARBURIZING

By L. D. Gable and E. S. Rowland

Abstract

This paper reviews the development of gas carburizing from its beginning to the present day and discusses modern practice in this art. Commercial installations for gas carburizing are described in detail from the standpoint of the various gases used and information given regarding their operation. Variables affecting case depth and character of the case are discussed and recommendations made regarding commercial limitations in the control of these variables.

Introduction

FOR the purposes of this paper alone, commercial gas carburizing is defined as those successful processes and practices in which the carburizing medium enters the retort in the gaseous state. The authors realize and fully appreciate the fact that all commercial carburization is fundamentally gaseous in nature and, indeed, is the reason for the limitations imposed in the above definition. Having once strayed afield from those processes in which the carburizing agent enters the retort as a gas, it is difficult to say what is and what is not gas carburizing. Hence, while realizing the commercial importance to those processes which utilize solid carburizing agents in conjunction with gases and those which use a liquid carburizing medium cracked to a gas, we will strictly limit ourselves in the present discussion to the definition as stated.

So much has already been published on the subject of gas carburizing that we cannot go into detail in reviewing the field. This published work cannot be dismissed, however, without paying tribute to the man most responsible for the development of the art and science of carburizing with gas — Federico Giolitti. Early work of scientific importance was published, beginning in 1860, by Caron (1)[1], Mannesmann (2) and others and later by Charpy (3), Guillet

[1]Numbers in parentheses refer to the appended bibliography.

A paper presented as part of the Symposium on Carburizing presented at the Nineteenth Annual Convention of the American Society for Metals held in Atlantic City, October 18 to 22, 1937. The authors are, respectively, metallurgist and research metallurgist with The Timken Roller Bearing Co., Canton, Ohio.

(4), Bruch (5) and others. It remained for Giolitti, however, in a series of papers published between 1908 and 1912, (6), (7), (8), to do much toward clarifying the vague generalities and differences of opinion of his predecessors. Working with carbon monoxide, ethylene, methane, and illuminating gas, he studied the effects of time, temperature and pressure on the depth of case obtained, the distribution of carbon in the case and its microscopic characteristics. Much of his work is still of value, but above all he provided the impetus for the commercial development of gas carburizing by the publication of his book (9) in 1912. In this work he brought together and critically reviewed all the known scientific work done up to that time and, in addition, explained in detail the then known commercial methods for gas carburizing.

Of the scientific work published in recent years on carburizing with gases, that of Bramley and his co-workers in England and of Sykes in this country deserves mention. In a series of papers published in the Carnegie Scholarship Memoirs of the Iron and Steel Institute from 1926 to 1929, Bramley and his associates (10), (11) studied carburizing power of carbon monoxide both wet and dry as well as the effects of time, temperature and rate of flow on this reaction. Their results showed that dry CO was much more effective than CO containing water vapor. Later work explored the carburizing action of CO + pyridine and CO + toluene in relation to the variables of time, temperature and rate of flow. Bramley also found that under his experimental conditions, periodic reversals of the flow of the carburizing gas were necessary in order to produce uniform carburizing. No evidence of the phenomenon of "liquation" as described by Giolitti was found, all curves of carbon content versus depth being smooth in the region of the eutectoid concentration. Cook (12) has critically reviewed Bramley's work from the standpoint of correlation with commercial practice and found several points of dissimilarity. The greatest discrepancy between Bramley's experiments and Cook's commercial experience lay in the question of the necessity of gas reversals for uniform carburization. Commercial practice dictates that reversal of flow is not necessary in properly designed and operated installations. Cook cites the following possible reasons for Bramley's results: (a) carburizing temperatures much higher than those used in commercial practice, thus magnifying any variations; (b) too low a rate of flow, particularly in regard to the experiments with

CO; and (c) the use of a silica muffle instead of the usual alloy muffle.

In a paper before the A.S.S.T., Sykes dealt with the gaseous carburization of Armco iron in mixtures of methane and hydrogen. He first explored the dissociation equilibrium conditions of methane at various temperatures. Sykes then determined the carburizing power of hydrogen saturated with methane at various temperatures and finally investigated the effect of time and increasing methane concentration. From this work he drew the following conclusions which are borne out by commercial experience: (a) For a given concentration of methane in hydrogen, the temperature which governs the rate of diffusion is the most important factor controlling the depth of case obtained. (b) At the higher carburizing temperatures, an increase in the concentration of methane results at first in greater case depth. Further increase in this concentration, however, does not increase the depth but only raises the carbon concentration at the surface. (c) If the gas has more than the minimum carburizing power at any given temperature, the rate of penetration of carbon is fixed by the rate of diffusion of the carbon in austenite which, in turn, is controlled by the carburizing temperature.

Development of Commercial Gas Carburizing

A brief description of the early work on gas carburizing pursued at The Timken Roller Bearing Company will serve to indicate the trend of development of commercial practice as well as the difficulties encountered.

Work was started early in 1913, soon after the results of Giolitti's investigations became available. It was apparent at that time that if Giolitti's results were correct and gas carburizing could be developed on a commercial scale, it would be an ideal method for carburizing roller bearing parts, due to better control of the character of the case, the elimination of packing small parts in compound and the dust and grime inevitably encountered when using solid carburizers. The availability of natural gas ideal for this purpose had much to do with the decision to begin work on this problem.

Consequently, a quite crude experimental apparatus was assembled to test the validity of Giolitti's published work. An old style, gas-fired, high speed tool furnace was used as a heating chamber.

The retort was composed of a heavy piece of cast iron pipe about 4 inches in diameter, 15 inches long and capped at both ends. The caps were drilled and tapped at their centers to accommodate lengths of ¼-inch pipe which served to carry the gas to and from the retort. Provision was also made for the entry of a protected platinum, platinum-rhodium thermocouple into the retort. Samples of bar stock 10 inches long and ⅜ inch in diameter were supported on racks in the retort. Most of the experiments were conducted on what are now S.A.E. 1015 and 5115 steels.

With this equipment, tests were run using natural gas, carbon monoxide and methane and the temperature range from 1500 to 1800 degrees Fahr. investigated in steps of 25 degrees Fahr. In addition, the influence of time and rate of flow on the case depth and character of the case were charted. It is not to be thought that these results were obtained without considerable difficulty. When the makeshift retort could be maintained gas-tight, quite satisfactory results were obtained although certain discrepancies appeared which were not explained until years later. As often as not, however, the cast iron pipe either burned through or leaks developed at the caps and the infiltration of air caused the specimens to decarburize as fast as they carburized—and sometimes faster.

During other experimental runs, the retort became so completely filled with soot that the flow of carburizing gas ceased of its own accord. During those runs in which soot formed, the retarding effect of soot on the rate of carburization was established.

Both solid carburizing compound and charcoal were tried in addition to the gases on several experimental runs. Peach pits were imported from California and tried with the gases because someone relayed the information that peach pits were all that was needed to make gas carburizing 100 per cent successful. That they were ever tried affords an illuminating commentary on the state of development of the science of metallurgy in 1913.

After some time, however, sufficient data were accumulated to convince the management that it was possible to carburize with the natural gas available at the plant. The problem of developing a furnace capable of carburizing on a commercial scale was immediately undertaken. The first unit built consisted of a steel shell, 15 feet high and 10 feet in diameter, heavily lined with firebrick and tangentially fired with gas burners. Inside this heating chamber a vertical retort about 3 feet in diameter was constructed of

firebrick, joined together with fireclay. This retort has a usable depth of about 12 feet, was solid masonry at the bottom except for the carburizing gas inlet and was fitted with a heavy steel cover at the top.

Numerous experimental runs were made on scrap bearing parts in this so-called commercial carburizing unit in 1914 and 1915 using natural gas with and without additions of charcoal and compound in the retort. Peach pits were given one last trial. The most serious difficulty encountered was infiltration of air into the retort through leaky joints. The retort was unable to withstand heating or accidental bumps during charging without cracking the fireclay mortar at one or more places. Due to the thickness of the retort walls, the heating time was excessive and the temperature variation inside the retort abnormally high. An average new retort performed satisfactorily for about the first two hours of the run when the fireclay between the bricks would begin to fall out. The usual procedure was to uncover the annular space between the retort and the outer wall from the top and try to patch the cracks with moist fireclay on a long handled paddle. This was generally unsuccessful but served to keep the test run limping along for a few more hours. Since air infiltration caused decarburization, case depth variations of 0 to 0.100 inch were often encountered.

The original problem of commercial gas carburization rapidly developed into a search for a gas-tight retort. Curved firebrick forms about 12 inches square by 3 inches thick with tongue and groove joints were obtained. A retort made from these forms showed no improvement over the first type as the fireclay in the joints cracked and it leaked air as badly as the former one. Next, firebrick cylinders, 3 feet in diameter, 2 feet high and about 3 inches thick were purchased and a retort built with the aid of the usual fireclay mortar. This retort did not leak quite as badly as the first two types but the cylinders broke easily under impact and were unable to withstand thermal changes. The next step was to build a smaller vertical furnace equipped with a one piece carborundum retort. This retort was practically gas tight but broke easily when bumped in charging and was generally too fragile for commercial use. A one piece firebrick retort was later installed and tried but it suffered from the same defects as it predecessor.

The final step in the early period of development was the purchase of a cast nichrome cylinder 18 inches in diameter, 3 feet long

and open at both ends. A furnace was built to accommodate this retort which was installed in a vertical position. The bottom of the retort was closed with a heavy iron casting, grooved to fit the end of the retort and lined with firebrick. The top of the retort was fitted with a brick lined dome-shaped cover. This retort did not break but was porous and leaked both through the body of the retort and at the joints on the ends. With this final failure, gas carburizing was abandoned as a commercial possibility. The World War intervened a few months afterwards and nothing further was done on the problem.

It can be seen from the foregoing that the long time interval between the experimental verification of the possibilities of carburizing with gas and its commercial success was due principally to the complete lack of a material suitable for a retort. When The Timken Roller Bearing Company again took up the problem in the spring of 1925, the retort problem was largely solved by the development of non-porous, machinable, one-piece retorts of heat resisting alloy. In addition, the familiar horizontal, rotary, batch-type carburizing machine had been developed. One machine was purchased for experimental purposes in May, 1925, and a battery of these units was installed for production operations in November of the same year. This type of machine essentially consists of a horizontal rotating alloy retort with a gas inlet at the center of the back end, the gas outlet being through a constricted opening in the center of the front cover. The retort rotates in a refractory lined steel shell which is mounted on trunnions for tilting. The equipment is designed for gas firing but it can be fired with oil if desired. A good description of the operation and advantages of this type of equipment has been given by O'Neil (14) and others. See Fig. 1.

From this time on, plant development progressed rapidly. In 1926, larger machines of the same type were put into operation. Over a period of the next few years many improvements were made in the original machines, such as correction of minor mechanical defects, development of better control valves and better burners, improved general design of retort and the development of retorts with internal longitudinal ribs to promote agitation of the work. In 1927, stationary vertical furnaces were installed for carburizing larger bearing parts. This type of furnace has a vertical retort with the gas inlet in the center of the bottom and a cover containing the gas outlet, which is firmly clamped on the retort. The annular com-

Fig. 1—A Battery of Horizontal Rotary Gas Carburizing Machines. Courtesy of American Gas Furnace Co.

bustion space between the retort and the refractory lined outer steel shell is tangentially fired by means of alloy tipped burners (Fig. 2). Operation of this furnace has been described by McNally (15) among others. Larger furnaces of this same type have recently been installed for carburizing still larger work. They differ from the above only in that they have four gas inlets in the bottom instead of one and that the retorts are of the annular type, having a circular flue in the center through which the combustion gases escape.

From 1927 to the present time, our development work has consisted mainly in the exploration of the commercial range of carburizing temperatures, the development of a proper ratio of carburizing to diffusion period, experimentation with the Selas gas mixing machine in the early stages of its development and extensive experimentation on the control of the carburizing process.

In 1931, Cowan (16) published the experimental results which led to the development of the Eutectrol process of continuous gas carburizing, a process which has since come to enjoy widespread application. Commercial installations have been described in detail by Heyn (17), Thomas (18) and Cowan (19). This unit consists essentially of a long, low, refractory lined steel shell which is both over and under fired. An alloy muffle, 31 inches wide and from 19 feet to 26 feet long, composed of flanged and bolted sections, runs through this furnace, the carburizing trays being pushed through the muffle on two sets of rollers and skids.

Carburizing gas is introduced at several points along the top of the muffle and at the charging end, the amount introduced at each point of entry depending upon the character of the case desired. At both ends of the furnace, large, air-operated valves effectively seal the muffle. Extensions of the muffle beyond these valves form two external chambers closed at their outer ends by lifting doors. The chamber at the charging end is used as a purging chamber in which all oxygen is expelled by means of flue gas. The corresponding chamber at the discharge end is used to produce any desired temperature of the work prior to quenching and provide a short diffusion period (Fig. 3).

The horizontal rotary machine previously described has been adapted for continuous carburizing by making a few changes. The retort is reduced in diameter, lengthened to about 6 feet and fitted with a spiral conveyor fin in addition to the longitudinal ribs.

Fig. 1—A Battery of Horizontal Rotary Gas Carburizing Machines. Courtesy of American Gas Furnace Co.

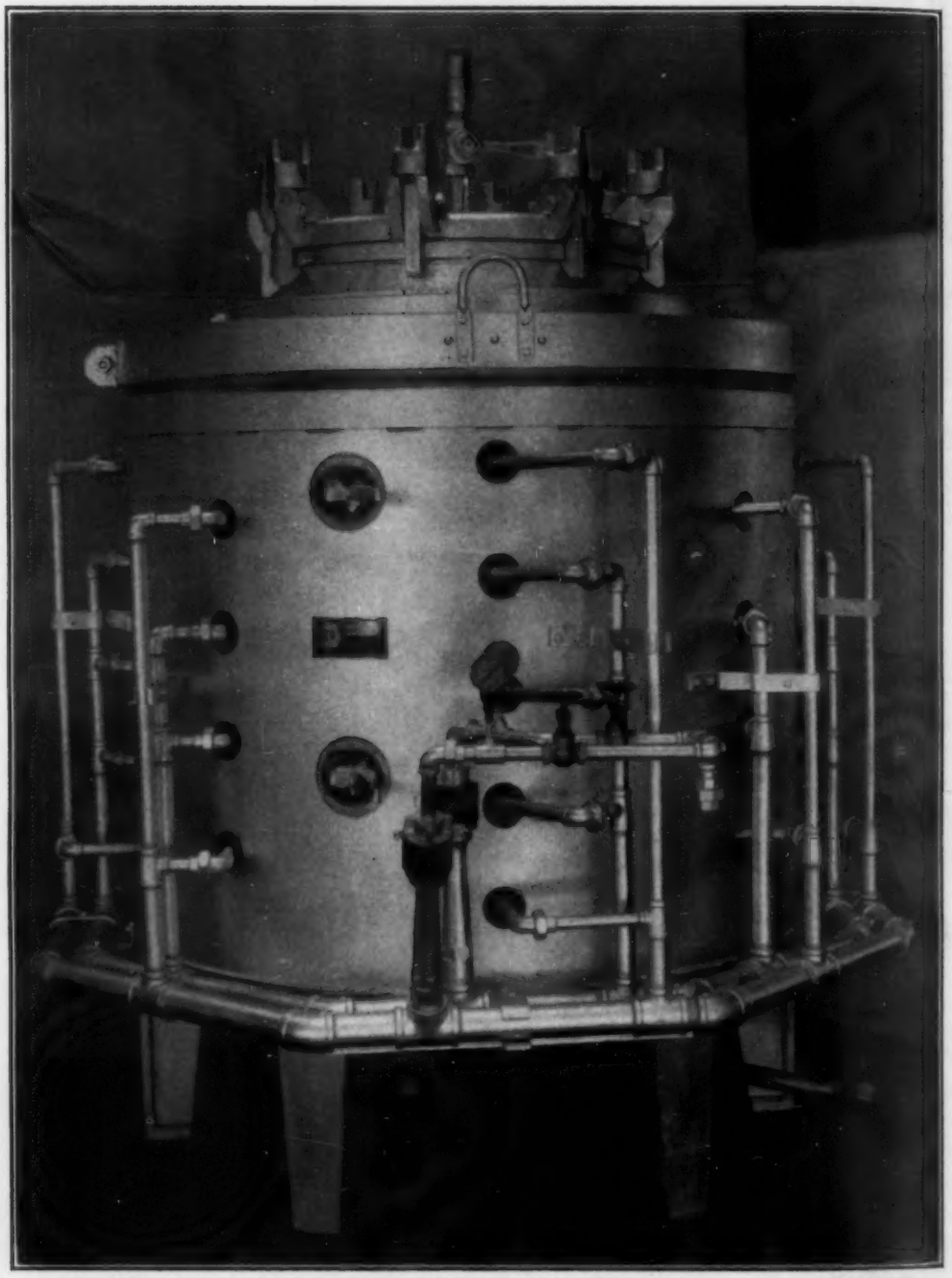

Fig. 2—Vertical Stationary Gas Carburizing Furnace. Courtesy of American Gas Furnace Co.

Carburizing gas enters at the center of the discharge end of the retort, travels through the retort counter current to the work and is vented through the top of the charging hopper. The design and operation of this type of continuous carburizer has been covered by Comstock (20). It is applicable principally to putting light cases on small parts.

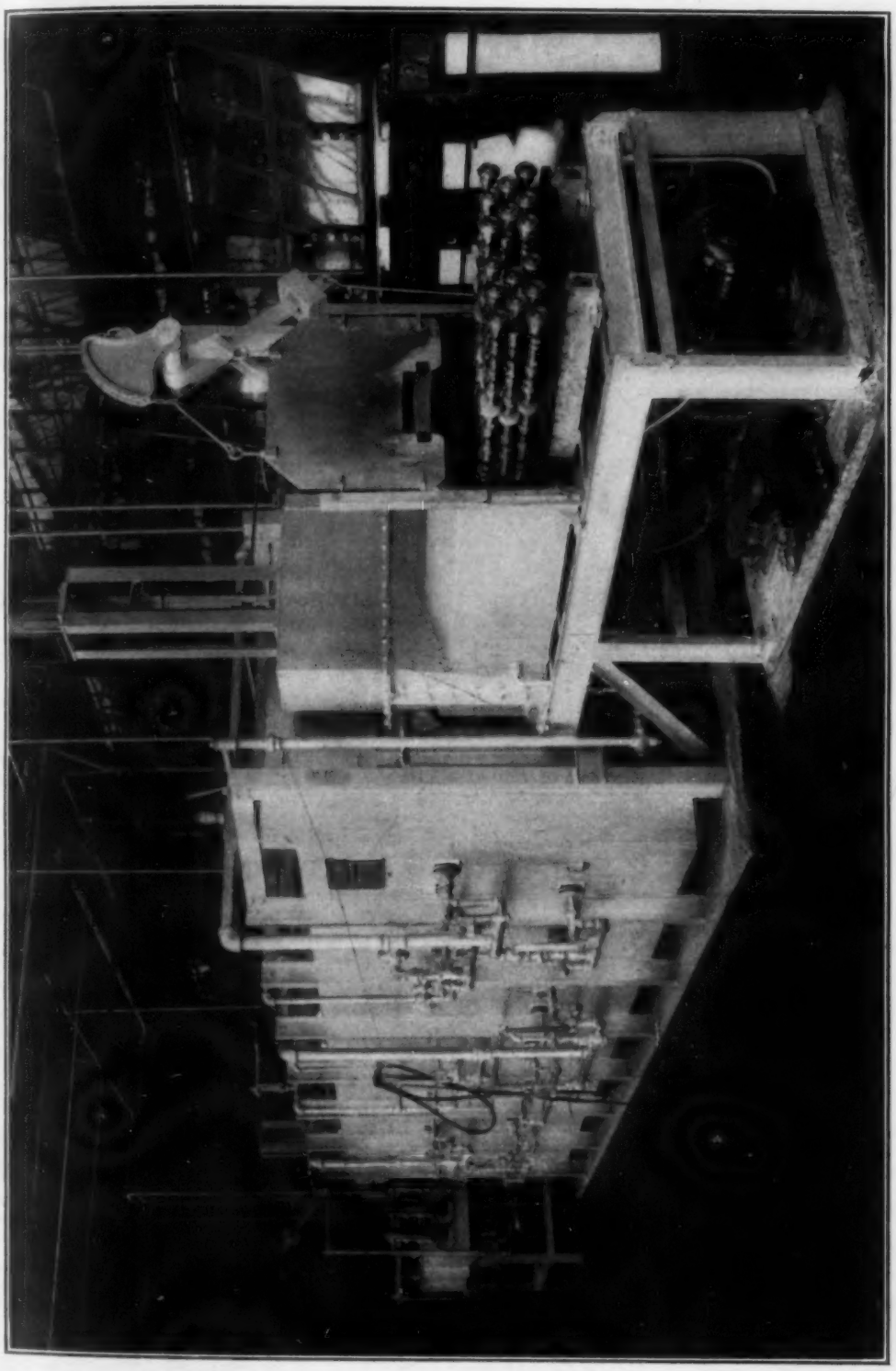

Fig. 3—A Continuous Gas Carburizing Furnace Operating on Butane. Courtesy of Surface Combustion Corp.

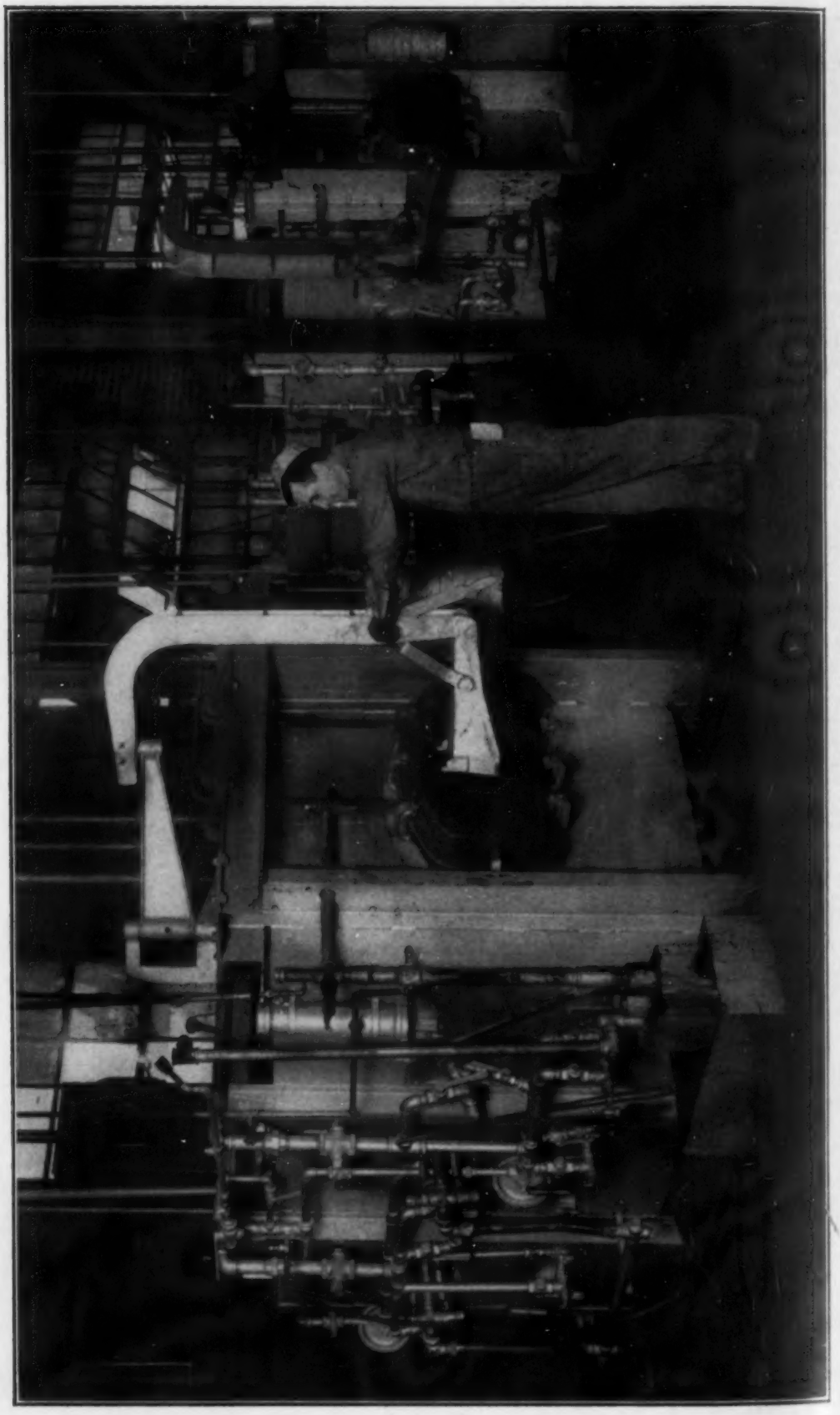

Fig. 4—A Pair of Box-type Batch Units for Gas Carburizing. Courtesy of Surface Combustion Corp.

A box-type, batch carburizing unit (Fig. 4) has been developed for the same applications as the cylindrical vertical furnaces. The brick lined shell contains a horizontal D-shaped muffle closed at the back end and fitted with a gas-tight door at the front. Carburizing gas enters through several places along the side of the muffle. The design and operation of these units has been described by Cone (21).

Modern Commercial Practice

It can safely be said that no two commercial installations for gas carburizing are exactly alike. Differences consist of variations in design of equipment, carburizing gases used, details of manipulation, type of steel used and character of case desired. These circumstances make discussion of present day practice difficult but, fortunately, certain fundamentals exist in all gas carburizing which serve to simplify the situation.

The gases being used at present for carburizing in commercial installations are: (a) natural gas, (b) propane, (c) butane and (d) manufactured or city gas. These gases may or may not be mixed with the following diluent gases depending upon conditions: (a) air and (b) prepared flue gas. City gas has not been successfully used as a diluent because of day by day variations in composition.

Natural gas may be used either raw or diluted with either of the above diluents. Propane and butane are usually diluted for use although at least one commercial installation has been operated with raw butane. Manufactured or city gas is invariably used in its undiluted state due to its low hydrocarbon concentration. In fact, city gas has been bubbled through benzol to increase its carburizing power but the authors do not know of any present installations utilizing this mixture. Difficulty is experienced at present in obtaining benzol or naphtha at a reasonable price and with a uniform specific gravity.

Typical analyses of gases now used for commercial gas carburizing are given in Table I, together with their calculated specific gravities and heating values in the majority of cases. Natural gas, either in the raw or diluted state, has been found satisfactory for carburizing, regardless of source. From any one source it has the advantage of being unusually constant in total hydrocarbon content and the disadvantage, if used in the raw state, of depositing excessive amounts of soot on the work and retort unless carefully han-

Fig. 4—A Pair of Box-type Batch Units for Gas Carburizing. Courtesy of Surface Combustion Corp.

Table 1
Typical Analyses and Properties of Commercial Gases

Type of Gas	Analysis—Per Cent by Volume CO_2	O_2	N_2	CO	H_2	Methane (CH_4)	Ethane (C_2H_6)	Ethylene (C_2H_4)	Benzene (C_6H_6)	Propane (C_3H_8)	Butane (C_4H_{10})	Specific Gravity	B.T.U. Per Cu. Ft. (Gross)
Natural Gas													
Birmingham, Ala.	....	....	5.0	....	....	90.0	5.0	....	....	....	....	0.60	1002
Pittsburgh, Pa.	....	....	0.8	....	....	83.4	15.8	....	....	....	....	0.61	1129
So. California	0.7	....	0.5	....	....	84.0	14.8	.. .	....	....	....	0.64	1116
Los Angeles, Cal.	6.5	....	....	....	....	77.5	16.0	....	....	....	....	0.70	1073
Kansas City, Mo.	0.8	....	8.4	....	....	84.1	6.7	....	....	....	....	0.63	974
Canton, Ohio	....	1.2	1.7	1.0	....	79.2	16.1	0.8	....	....	....	0.655	1109
Columbus, Ohio	....	0.2	1.5	....	....	80.1	18.0	0.2	....	....	....	0.65	1132
Cleveland, Ohio	....	0.6	0.5	0.8	....	80.7	17.0	....	....	....	....	....	....
Mixed Gas													
Natural & Water Gas	4.4	2.1	4.7	25.5	35.1	23.1	4.7	0.2	0.2	....	....	0.61	525
Manufactured Gas													
Coke Oven Gas	2.2	0.8	8.1	6.3	46.5	32.1	....	3.5	0.5	....	....	0.44	574
Carburetted Water Gas	1.8	1.6	13.6	9.0	42.6	28.0	....	2.4	1.0	....	....	0.50	528
City Gas													
Toledo, Ohio	2.8	0.3	3.3	8.4	48.1	33.1	....	....	....	....	....	....	....
Chicago, Ill.	1.1	1.5	5.5	5.6	22.0	64.3	....	....	....	....	....	....	....
Syracuse, N. Y.	3.0	0.6	5.6	12.1	44.6	29.8	....	....	....	....	....	....	....
Commercial Butane	....	....	....	....	....	....	....	....	....	7.0	93.0	1.95	3225
Commercial Propane	....	....	....	....	....	....	....	....	....	100.0	....	1.52	2572

Table II
Data on Commercial Gas Carburizing Installations

Type of Furnace	Part Carburized	Gas Used	Type Steel	Case Depth Total Penetration	Total Time Cycle	Diffusion Period
Box-type Batch	Side Gears	Natural + DX	3115	0.045″	6 hrs.	none
Box-type Batch	Bearing Races	Natural + DX	4615	0.080″	10 hrs.	none
Box-type Batch	Piston Pins	Natural + DX	1020	0.045″	5½ hrs.	none
Box-type Batch	Clutch Parts	Raw Nat. & Nat. + DX	4620	0.030″	3 hrs.	none
Box-type Batch	Camshafts	Propane + CG	1020	0.070″	8½ hrs.	none
Horizontal Rotary	Bearing Parts	Raw Natural	4620	0.040″ to 0.080″	4½ to 15 hrs.	1½ to 5 hrs.
Horizontal Rotary	Pins	Raw Natural	1020	0.040″ to 0.125″	4 to 25 hrs.	none
Horizontal Rotary	Bearing Parts	Propane + CG	4620	0.050″ to 0.120″	5½ to 19 hrs.	none
Horizontal Rotary	No Data	Raw Butane	No data	0.035″	4 hrs.	none
Stationary Vertical	Bearing Parts	Raw Natural	Krupp	0.070″ to 0.210″	15 to 105 hrs.	5 to 35 hrs.
Stationary Vertical	Bearing Parts	Propane + CG	4620	0.100″ to 0.150″	27 to 37 hrs.	2 hrs.
Stationary Vertical	Shafts	Raw Natural	1020	0.060″	7 hrs.	2 hrs.
Continuous	Plowshares	Butane + CG	1020	0.080″	10 hrs.	none
Continuous	Bearing Races	Propane + CG	4615	0.070″	10 hrs.	none
Continuous	Ring Gears	Natural + DX	4615	0.060″	7½ hrs.	none
Continuous	Camshafts	Propane + CG	4620	0.060″	7½ hrs.	none
Continuous	Ring Gears	Natural + CG	4615	0.045″	6 hrs.	none

dled. The heavy hydrocarbons—propane and butane—have the disadvantage of depositing a tarry substance which cokes on the work and causes spotty carburization unless diluted or carefully handled within a narrow range of temperature. While the above gases are used in most commercial work, manufactured gas is used to some extent. In some cases it can be successfully used without treatment but where nonuniform results are obtained, drying the gas to a low and constant water vapor content frequently eliminates further trouble.

Present day commercial gas carburizing practice will be discussed from the standpoint of the gases used under the following headings:

A. Raw natural gas
B. Diluted natural gas
C. Raw butane
D. Diluted propane or butane
E. Manufactured or city gas
F. Miscellaneous

Data on a large number of commercial installations have been collected in Table II. Information regarding the type of equipment, parts carburized, the carburizing medium, type of steel, case depth obtained (total penetration) and the time at carburizing temperature (1650 to 1750 degrees Fahr.) is included.

A. Raw Natural Gas

The use of raw natural gas is entirely confined to batch type operation. As an example, we will describe our own practice in some detail. Analyses of the steels used in making Timken roller bearing parts are given in Table III.

Table III
Analyses of Steels Used

Type	C	Mn	P	S	Si	Cr	Ni	Mo
S.A.E. 4620	.18/.22	.45/.55	.040 Max.	.040 Max.	.23/.28	.30 Max.	1.65/1.85	.21/.25
Krupp	.12/.15	.35/.45	.025 Max.	.025 Max.	.25/.30	1.35/1.55	3.85/4.25	Nil

Natural gas of the analysis shown in Table I is prepared by a simple purification treatment in a linseed oil scrubber. The carburizing gas is bubbled through about 2 feet of linseed oil in either of the two large scrubbers provided. The only function of the

scrubber is to remove foreign matter such as dirt, pipe scale and tar deposits. Extensive experiments on purification have been carried out in the past using cotton, calcium chloride, benzene, etc., as purifying agents but the above practice is all that has been found necessary for successful use of the gas.

The scrubbed gas is delivered to each carburizing furnace under a pressure of 6 ounces per square inch. Rate of flow to each furnace is regulated by placing a choker in the inlet pipe. The choker is a thin metal disk with a $\frac{5}{32}$-inch diameter orifice in its center. This is placed in a pipe union and passes 90 cubic feet of gas per hour at the above line pressure. On the large vertical furnaces, 90 cubic feet per hour enters through each of the 4 inlets. Some operators of this type of equipment prefer flow meters and regulators but our gas pressure is sufficiently constant to make the above arrangement entirely satisfactory. All outlet pipes from the retort are fitted with a $\frac{5}{16}$-inch diameter orifice, which maintains the gas under a slight pressure in the retort. This orifice tends to become plugged with soot and scale, and to assure uniform operating conditions, is cleaned once each hour. For this purpose, a $\frac{1}{4}$-inch diameter rod, flattened on the end to just under $\frac{5}{16}$ inch, is inserted in the orifice with a twisting motion.

A carburizing temperature of 1650 degrees Fahr. for Krupp and 1700 degrees Fahr. for S.A.E. 4620 steel is maintained within plus or minus 10 degrees Fahr., in any one charge, from charge to charge and among the various furnaces as well. This small spread in work temperature is maintained only at the price of eternal vigilance. Control thermocouples are placed in the combustion space to reduce temperature lag in operating; hence the control temperature is well above the retort and work temperature. Thermocouples and control instruments are checked at short intervals; all charges are checked for temperature before carburizing is begun by inserting a thermocouple through the outlet pipe; and the temperatures of all furnaces are measured with an optical pyrometer at least once in each 8-hour turn.

The rotary retorts are charged with a constant volume of material, regardless of the size of the parts, except for a set maximum size for each type of furnace. This is accomplished by the use of a standard size of charging box which is filled to a constant level. A charge of two-thirds of the retort volume has been found to give best results from the standpoint of tumbling of the work in the

Table IV
Data on Furnace Charging

Type of Furnace	Retort Vol. Cu. in.	Size of Charges	Max. Size of Parts	Time Allowed for Charge to Reach Temp.
Small rotary	9,000	⅔ volume	2" dia. x 2" long	1½ hours
Large rotary	18,500	⅔ volume	4" dia. x 3" long	2 hours
Small vertical	21,000	1300 lbs.	28" O.D. x 28" long	4 hours
Large vertical	70,000	4000 lbs.	47" O.D. x 40" long	6 hours

retort. Since the ratio between volume and weight of bearing parts is nearly constant, this volume corresponds to about 600 and 1200 pounds of material for the small and large rotary machines, respectively. In the vertical furnaces, standard sizes and shapes of fixtures are used to hold the work and a constant weight of material is charged. Data on the sizes of retorts, size of charge, and the time allowed to heat the charge to the carburizing temperature are given in Table IV. All retorts are at the carburizing temperature before charging is begun.

The sequence of our operations in carburizing has been standardized with but few exceptions, regardless of the type of furnace. With the retort at temperature, the definite charge is placed in the furnace together with standard carburizing test rings of the same type of steel as the charge. From two to six rings are spaced through the charge and are later analyzed (Fig. 5A). Gas-tight covers are securely fastened and the gas passed through the retort for 5 minutes in order to purge the retort of air. The gas outlet is then plugged with a tapered pin, the gas shut off and the charge allowed to come to the temperature for the period given in Table IV. Temperature of the work is then checked and the gas turned on for the duration of the carburizing cycle. The gas outlet is again plugged, the gas turned off and the charge held at the carburizing temperature for the diffusion cycle, a period half as long

Table V
Carburizing Times Required for Various Case Depths

Case Depth In.	Type of Steel	Carb. Temp.	Carburizing Time in Hours		
			Gas	Diffusion	Total
0.030	S.A.E. 4620	1700	3	1½	4½
0.045	S.A.E. 4620	1700	5	2½	7½
0.070	S.A.E. 4620	1700	14	7	21
0.060	Krupp	1650	10	5	15
0.075	Krupp	1650	18	9	27
0.100	Krupp	1650	28	14	42
0.125	Krupp	1650	48	24	72
0.185	Krupp	1650	70	35	105

as the carburizing time. Times required to produce various case depths on both Krupp and S.A.E. 4620 steel by this practice are given in Table V. In the large vertical furnaces where deep cases are produced on large parts and the diffusion periods are long, a gas period of 5 minutes at the standard rate is employed midway in the diffusion cycle as a safeguard against slight air infiltration.

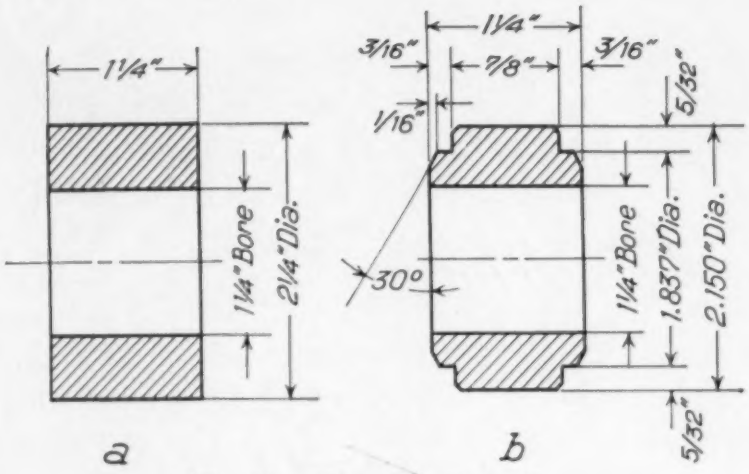

Fig. 5A—Carburizing Test Ring.
Fig. 5B—Carburizing Test Ring After Machining.

When the cycle is completed samples of the carburized material are taken from both ends and the middle of the charge, quenched in water and fractured for case depth measurement. Such fractures are etched in a solution of 20 per cent nitric acid in water and the light shaded area of the case is measured as the case depth. This depth corresponds to a point where the carbon content is between 0.40 and 0.50 per cent as we feel that the benefits of carburizing are obtained only on this portion of the case. If the measured depth is satisfactory, the carburizing test rings are removed, cooled in mica and the charge quenched in oil.

Test rings are machined on each end a distance equal to twice the case depth (see Fig. 5B). They are then cleaned on the O.D. and 10 cuts, each of 0.005 inch depth, machined and the turnings analyzed for carbon. In this way, accurate information is obtained on the maximum carbon content in the case and on the carbon gradation to a depth of 0.050 inch. Our standard practice is to hold carbon content in the case between 0.85 and 0.95 per cent on finished bearing parts. Typical analytical results are given in Table VI. It must be remembered that the minimum grinding allowance

Table VI

Data on Carbon Concentration Through Case

Type Furnace	Type of Steel	Carb. Temp. Degrees Fahr.	Carb. Gas	Cycle Hrs. Diff.	Case Depth In.	Carbon Content in Per Cent Between 0 and 0.050 Inch Depth									
						1	2	3	4	5	6	7	8	9	10
Small Vertical	Krupp	1650	70	35	0.185	1.04	1.01	0.93	0.90	0.89	0.86	0.85	0.83	0.82	0.78
Small Vertical	Krupp	1650	70	35	0.185	1.03	1.00	0.93	0.90	0.88	0.87	0.85	0.84	0.82	0.78
Large Vertical	Krupp	1650	70	35	0.185	0.99	0.96	0.92	0.90	0.86	0.82	0.78	0.75	0.71	0.68
Small Vertical	Krupp	1650	12	6	0.065	0.94	0.92	0.87	0.86	0.80	0.76	0.70	0.65	0.60	0.55
Small Vertical	Krupp	1650	12	6	0.065	0.93	0.90	0.89	0.85	0.79	0.73	0.67	0.61	0.55	0.49
Small Vertical	4620	1700	10	5	0.065	0.98	0.96	0.90	0.87	0.82	0.78	0.74	0.70	0.66	0.63
Small Vertical	4620	1700	10	5	0.065	0.98	0.96	0.93	0.90	0.86	0.82	0.78	0.75	0.72	0.68
Small Rotary	4620	1700	3	1½	0.030	0.96	0.93	0.79	0.71	0.62	0.55	0.47	0.37	0.30	0.29
Small Rotary	4620	1700	3	1½	0.030	0.97	0.96	0.90	0.87	0.75	0.65	0.51	0.42	0.34	0.26

Note—All data are taken from different charges on different furnaces.

is 0.005 inch on the radius and greater on the larger parts carrying deeper cases.

In addition to test ring analyses, longitudinal sections from about 10 per cent of our fracture tests are ground on a fine belt grinder, etched for one minute in a 0.6 per cent solution of nitric acid in water and the case depth measured with a Brinell glass. Numerous experiments have shown that the point where the color changes abruptly from blue to brownish gray corresponds to between 0.50 and 0.45 per cent carbon. On thinner cases, this depth is from 0.010 to 0.015 inch less than the total case depth and for deep cases is from 0.020 to 0.025 inch less than the actual full penetration of carbon. This serves as a more accurate check on the case depth read from the rough fractures.

B. Diluted Natural Gas

The same control of carbon gradation and maximum carbon concentration in the case can be obtained by properly diluting natural gas and eliminating a no-gas or diffusion period, as is produced by using raw natural gas with a subsequent diffusion period. The above factors are controlled by regulating the ratio of volume of natural gas to volume of diluent gas. In rotary machines and vertical furnaces, natural gas is usually diluted by mixing with air in a mixing machine such as the Selas mixer. For box type batch operation or continuous furnaces, a DX unit is used to prepare burned gas of constant composition from natural gas (See Fig. 6). In this unit, practically perfect combustion of the air and natural gas occurs and a flue gas of almost any desired composition can be produced.

In a typical box type batch installation, DX gas containing 8 per cent CO, 9 per cent H_2 and 4.5 per cent CO_2 is mixed with raw natural gas in the proportion of 40 cubic feet of natural gas to 60 cubic feet of DX gas per hour and fed to the muffle under a pressure of 1 inch of water. Gears made from S.A.E. 3115 steel are given a 0.045-inch case in 6 hours at 1700 degrees Fahr. A variation of this type of practice consists of using raw natural gas for part of the carburizing cycle followed by a mixture of raw natural gas and DX gas for the balance of the time period.

Continuous furnace practice usually involves the use of DX gas containing from 1 to 3 per cent CO mixed with natural gas at a muffle pressure of 1 inch of water with a total flow of between 150

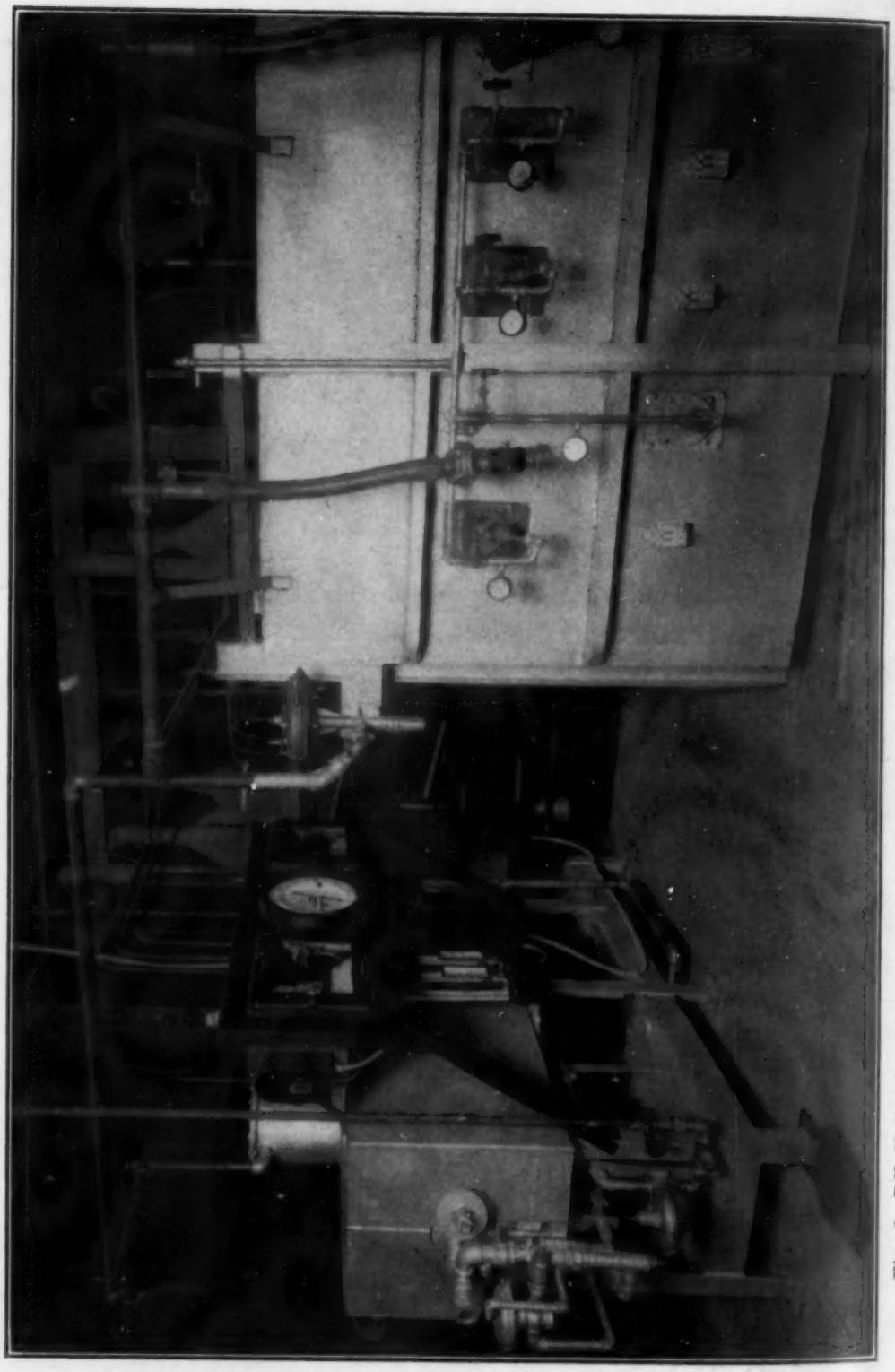

Fig. 6—DX Unit (At Left) for Preparing Flue Gas of Constant Composition. Courtesy of Surface Combustion Corp.

and 250 cubic feet per hour. The total flow used depends primarily upon the size and shape of the pieces being carburized. Too low a rate of flow results in slow carburization while too high a flow produces nonuniformity because of turbulence. The proportion of raw gas to DX gas used depends upon the character of the case desired. On carbon and low alloy steel parts of medium size carburized at 1700 degrees Fahr., a 0.100-inch hypereutectoid case (1.15 per cent carbon max. with 50 per cent of eutectoid composition or higher) requires about 60 per cent raw natural gas and 40 per cent DX gas at a total flow of 150 cubic feet per hour. A eutectoid case of the same depth requires a 1:1 ratio of natural gas to DX gas at the same rate of flow. Times are figured on the basis of 0.008 inch penetration per hour. Gas inlets are located at the charging end and at several points along the top of the muffle. Natural gas is mixed with the DX gas at the furnace and the proportion of natural gas mixed at each inlet increases toward the discharge end of the muffle to constantly enrich the gas traveling through the retort. The proportions given above are totals for all inlets.

One continuous installation in operation at present uses a CG unit instead of the customary DX unit to prepare the diluent gas. In this unit (Fig. 7) a mixture of 70 cubic feet of air and 50 cubic feet of natural gas per hour is externally heated to 1840 degrees Fahr., cracking the natural gas to give a product of constant composition. The total flow of CG and natural gas to the muffle is 224 cubic feet per hour, of which 120 cubic feet is CG gas and 104 cubic feet is natural gas. This total flow of natural gas is added at each inlet in varying proportions. Ring gears of S.A.E. 4615 steel, weighing 21 pounds each, are carburized to a case depth of 0.045 inch (total penetration) at 1750 degrees Fahr., the daily production being 384 gears. The total time in the furnace is 6 hours. Raw natural gas is introduced in the holding chamber at the discharge end to prevent decarburization.

C. Raw Butane

The only installation using the heavy hydrocarbons without dilution, of which the authors are aware, has been described by Spicer (22). Raw butane was used as the carburizing medium in an 800-pound capacity rotary machine. A case depth of 0.035 to 0.040 inch was obtained at about 1650 degrees Fahr. using a gas flow of 15 cubic feet per hour for the first two hours and 8 to 10 cubic

Fig. 6—DX Unit (At Left) for Preparing Flue Gas of Constant Composition. Courtesy of Surface Combustion Corp.

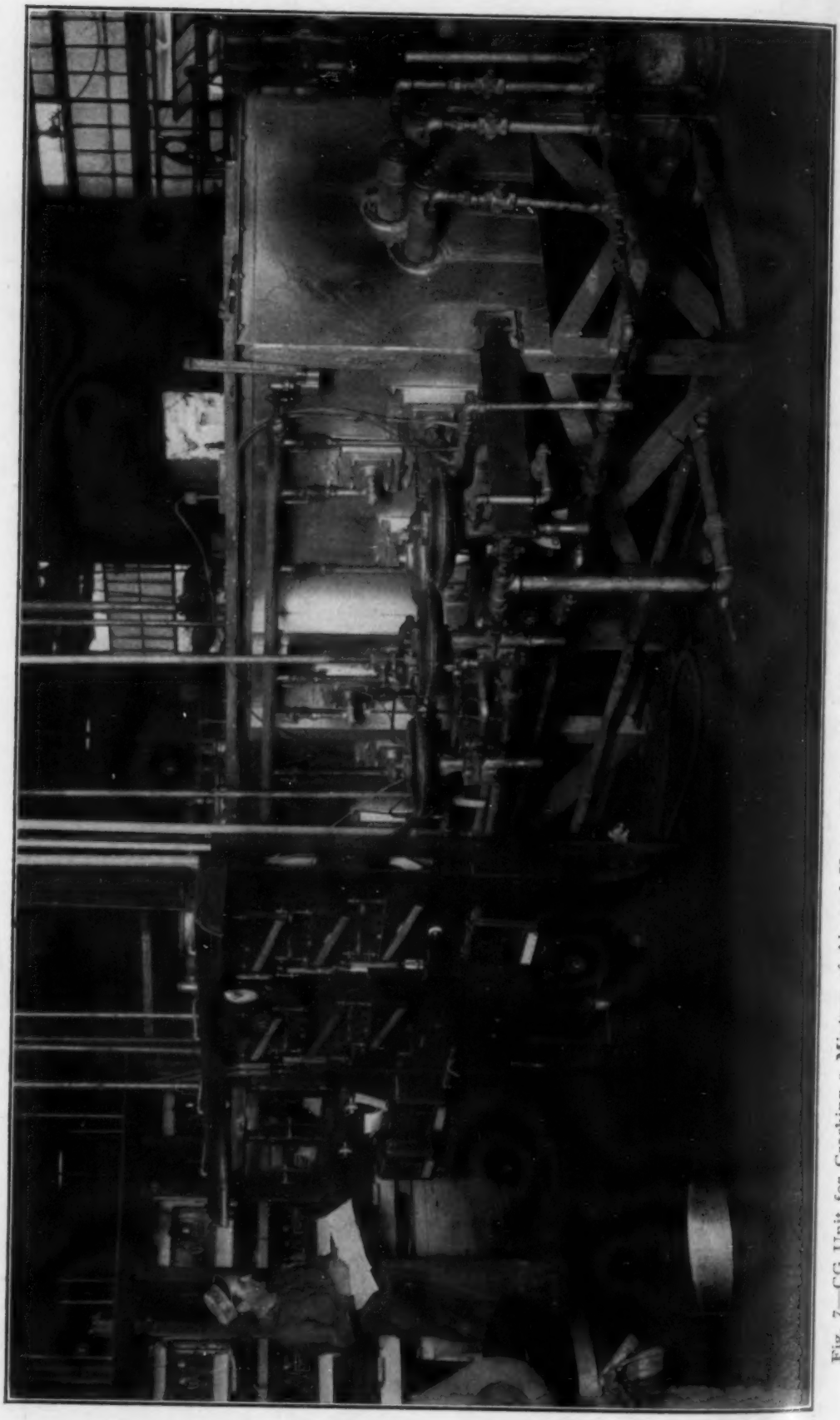

Fig. 7—CG Unit for Cracking a Mixture of Air and Heavy Hydrocarbon Gas at High Temperatures. Courtesy of Surface Combustion Corp.

feet per hour for the remaining two hours. No difficulty from tar or soot was experienced and no advantage was found in mixing other gases with the butane or in surging.

D. Diluted Propane and Butane

Practically all commercial installations utilizing propane or butane as the carburizing medium also use some method of diluting these gases. For rotary machines and vertical furnaces, they are generally mixed with air in a mixing machine. For continuous and box type batch furnaces, a CG unit is used to prepare a cracked gas for mixing purposes. In this unit the hydrocarbon gas is diluted with air in the proportions of 1 part propane to 7 parts of air or 1 part butane to 10 parts of air and externally heated to a higher temperature than the carburizing temperature in order to crack the hydrocarbons without the formation of tar or soot. This gas is mixed with propane or butane at the inlets in the same way as when diluted natural gas is used. The proportions, of course, are different. Cowan (23) has recently published some observations and experimental work on continuous furnaces using these gases.

One typical installation of stationary vertical and horizontal rotary furnaces uses a Selas mixed gas composed of one part by volume of propane and 2 parts by volume of air in carburizing S.A.E. 1015 and 4620 steels at 1700 degrees Fahr. Large parts are carburized in the vertical furnaces, using the following cycle of operations: With the furnace at temperature the charge is introduced and the gas, flowing at a rate of 100 cubic feet per hour, is passed through the retort for 10 minutes to purge the muffle of air. The gas is then turned off for 3 hours to permit the charge to come to temperature and again turned on for the carburizing cycle. A diffusion period of 2 hours is allowed, regardless of the length of

Table VII
Case Depth vs. Time for 1:2 Propane-Air Mixtures

Case Depth In.	Type of Furnace	Carburizing Time at 1700 Degrees Fahr. Gas	Diffusion	Total
0.050	Rotary	5½	.	5½
0.070	Rotary	7½	.	7½
0.100	Rotary	15½	.	15½
0.120	Rotary	19	.	19
0.100–0.115	Vertical	25	2	27
0.115–0.130	Vertical	29	2	29
0.125–0.140	Vertical	31	2	33
0.135–0.150	Vertical	35	2	37

Fig. 7—CG Unit for Cracking a Mixture of Air and Heavy Hydrocarbon Gas at High Temperatures. Courtesy of Surface Combustion Corp.

the carburizing cycle. In the rotary machine the same gas is used at a flow of 65 cubic feet per hour and the same cycle of operations employed except that only 1 hour is allowed for the charge to reach the carburizing temperature and that no diffusion period is used. The carburizing and diffusion times (if any) for definite case depths are given in Table VII. These depths are total carbon penetration and account for much of the discrepancy between Tables V and VII.

A typical continuous furnace in operation at present uses butane and air, cracked in a CG unit, as the base gas and raw propane as the enriching gas for carburizing medium sized parts of S.A.E. 1015 and S.A.E. 4620 steel at 1700 degrees Fahr. A mixture of 140 cubic feet of air and 15.5 cubic feet of butane per hour is fed to the CG unit and partially burned or cracked at 1760 degrees Fahr. This gas is cooled by passage through a water spray in a closed tower and then dried by means of a calcium chloride spray of 1.4 to 1.45 degrees Baumé gravity. Finally the gas is passed through a tower packed with excelsior and fed to the furnace. A total amount of 50 cubic feet per hour of propane is mixed with this gas at the various inlets. The total time cycles required for various case depths (total penetration) are given in Table VIII and include heating time as well as a short period in the discharge chamber to allow the work to equalize at a temperature of 1500 degrees Fahr. before quenching.

Table VIII
Case Depth vs. Time Cycle in Continuous Furnace

Case Depth, In.	Total Time Cycle Hours
0.055–0.070	10
0.065–0.080	11
0.080–0.095	14
0.100–0.115	17
0.110–0.125	20

E. Manufactured or City Gas

Where natural gas is available it is universally used for gas carburizing. In plants where it is not available, the choice lies between propane or butane and city gas. In all but a very few cases, the former have been chosen. The principal disadvantages of manufactured gas are a low content of hydrocarbons, necessitating compression of the gas to pack sufficient active agent into the retort at

one time, and considerable variation in the hydrocarbon concentration from time to time because city gas is produced on a constant B.t.u. basis. Carburization under pressure causes operating hazards due to leakage, etc., while the variation in analysis renders duplication of results difficult.

Guthrie and Wozasek (24) have published some results of experimental carburization with city gas of an analysis very close to that given in Table I as from Chicago, Ill. Their results showed that some of the variation in results was due to varying water vapor content of the gas. Commercial operation with city gas dried to a constant small content of water vapor has substantiated their experiments. Dried gas did not produce entirely consistent results, however, and further investigation led them to believe that for uniform results a small amount of oxygen in the gas was necessary at the beginning of carburization to produce an oxide film on the metal which acted as a catalyst. Guthrie (25) proposed the addition of 10 drops of water per minute to the dried gas (saturated at 32 degrees Fahr.) for the first 6 minutes of the cycle to produce this oxide catalyst which he stated would prevent excess soot formation even at the high pressure used (15 pounds per square inch) and produce consistent results on a par with those produced with other gases.

F. Miscellaneous

Wilbor and Comstock (25) proposed the intermittent surge method of gas carburizing in 1930. This method consisted of surging heavy hydrocarbon gases over the work in a batch type retort followed by alternate surges of oxidizing gas which served to remove a thin film of "dead" gas on the surface of the work and prepare the latter for the next surge of hydrocarbon gas. A typical cycle of operation, as stated by Guthrie (24) is as follows: A given volume of heavy dry hydrocarbon gas at 7 to 15 pounds pressure is introduced into the retort for from 5 to 15 seconds and the gas valves closed for from 5 to 10 minutes while carburizing proceeds. This cycle is repeated 3 times with a 5 to 10-minute stand-by between surges. Then 5 per cent of CO_2 is introduced in the same way for one cycle, clearing the retort of residual hydrocarbon gas and reactivating the metal surface. Then surges of the hydrocarbon gas are again begun, followed in turn by a surge of CO_2 gas. The entire cycle is repeated until the desired case depth is obtained. This method of gas carburizing is obviously complicated and commercial

practice has shown that the same results can be produced by dilution to a constant analysis of mixed gas and a steady flow of this gas. The authors are aware of no present commercial installations utilizing the intermittent surge method.

Control of the Process

Modern gas carburizing furnaces and machines are well designed units in which carburizing of high quality and uniformity can be done at low cost and in the shortest possible time. Control of the process, therefore, consists of producing, within commercial limits, a constant depth of case, a uniform maximum concentration of carbon at the surface and a satisfactory gradation in carbon from case to core. Gas carburizing is a ticklish operation at best and simplicity in a process is a virtue not to be underrated. Many variables enter in, some controllable and others not, but the fewer the total number of points in the process where variations may possibly occur, the easier will be the problem of control and the better the results obtained.

The most important variables entering into the process are (a) temperature, (b) the gas, (c) the time, (d) the steel, and (e) the human element. Of these variables, temperature is the most important because, in commercial operation, the entire process is dependent upon the rate of diffusion of carbon in austenite which, in turn, is dependent on the temperature. The necessity for the most rigid control of the carburizing temperature cannot be too heavily stressed. It is the author's opinion that the temperature must be held within a $\pm$ 10-degree Fahr. variation if best results are to be obtained. This figure includes variation through the charge as well as from charge to charge in the same furnace and from one furnace to another operating on the same product. This small variation can only be maintained by continual checking and adjustment of the equipment but it is felt that the effort will be repaid in results.

Variables due to the gas include composition and rate of flow. Both should be carefully measured and held constant within as narrow limits as possible. Pressure affects the rate of flow and should be held constant for this reason. Gas pressure in the retort should be sufficient to prevent infiltration of air. Increasing the pressure beyond this point, however, is of no benefit unless the gas is so dilute that the rate of transfer of carbon to the steel is less than the rate of diffusion. Under these conditions, compressing the gas is

necessary to increase the amount of hydrocarbon gas in the retort at any one time. The obvious solution, however, is to increase the hydrocarbon concentration of the gas used and operate at a lower pressure, where operating difficulties are lessened.

The effect of variation of time on the depth of case is much less important than that of temperature variation but it is of sufficient importance that the time cycle of operations must be rigidly adhered to. The more complex the cycle, the more difficult of attainment is the control of this variable.

Both the analysis of the steel and the size of the austenite grains at the carburizing temperature have an effect on the case obtained. In general, of course, the more highly alloyed the steel the slower the rate of carbon diffusion. Variations within the ordinary limits of analysis, however, produce some differences in the resultant case. Generally speaking, the larger the austenitic grain size, the greater the rate of penetration of carbon. The need for control of these variables is usually greater from the standpoint of the finished part than from that of the carburizing operation, but their effects in this regard should not be overlooked.

The human element enters into the control of all the above variables. Any operating man is kept painfully aware of the effect that the human element has on the process he is trying to control. This factor cannot be entirely eliminated but proper training and discipline will do much toward minimizing it.

Controversy often arises as to the commercial limits of case depth within which the product of a properly operated process can be maintained over a long period of time. It is the authors' belief that if carburization is carried out by the best commercial practice available, the variation in case depth should not exceed $\pm$ 0.010 inch (for case depths of 0.030 inch or greater) but that it cannot be maintained within much less than this variation over long periods. For example, if a part has a specified case depth of 0.060 inch, a minimum of 0.050 inch and a maximum of 0.070 inch should be acceptable and the case depth of all parts maintained within these limits.

This statement is made only after years of careful checking of operating conditions and the fracturing and measuring of numerous entire charges of large commercial gas carburizing furnaces, a procedure which often entailed the measuring of 1500 pieces per charge. It is entirely possible that a single furnace charge can be carburized

to within closer limits of case depth but, from charge to charge and furnace to furnace, the best commercial practice in use today still produces a case depth variation of ± 0.010 inch.

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Bibliography

1. Caron, *Comptes Rendus,* 1860, 2nd sem. p. 565
2. Mannesmann, *Verhandlungen des Vereins zur Beförderung des Gewerbfleisses,* 1879, Vol. 58, p. 31
3. Charpy, *Comptes Rendus,* 1903, 1st sem. Vol. 136, p. 1000
4. Guillet, *Iron and Steel Magazine,* 1904, Vol. 8, No. 4, p. 309
5. Bruch, *Metallurgie,* 1906, Vol. 3, p. 123
6. Giolitti and Carnevali, *Gazz. Chimica Italiana,* 1908, Vol. 38, Part 2, p. 309
7. Giolitti and Tavanti, *Gazz. Chimica Italiana,* 1909, Vol. 39, Part 2
8. Giolitti and Astorri, *Gazz. Chimica Italiana,* 1910, Vol. 40, p. 1
9. Giolitti, "The Cementation of Iron and Steel." Translated by Richards and Rouiller. McGraw-Hill Book Co., 1915
10. Bramley and Jinkings, Iron and Steel Inst. Car. Schol. Mem., 1926, Vol. 15, p. 17
11. Bramley and Lawton, Iron and Steel Inst. Car. Schol. Mem., 1927, Vol. 16, p. 35
12. Cook, *Heat Treating and Forging,* 1932, Vol. 18, Sept., p. 531, Oct., p. 588 and Nov., p. 642
13. Sykes, Transactions, American Society for Steel Treating, 1927, Vol. 12, No. 5, p. 737
14. O'Neil, *The Iron Age,* 1926, Nov. 18
15. McNally, Metal Progress, 1931, Vol. 19, Jan., p. 39
16. Cowan, American Institute of Mining and Metallurgical Engineers, 1931, Iron and Steel Div., Vol. 95, p. 386
17. Heyn, *Steel,* 1933, Vol. 92., Jan. 9, p. 17
18. Thomas, *Metals and Alloys,* 1932, Vol. 3, April, p. 84
19. Cowan, Metal Progress, 1933, Vol. 24, Oct., p. 44
20. Comstock, Metal Progress, 1936, Vol. 29, No. 4, April, p. 44
21. Cone, *Metals and Alloys,* 1936, Vol. 7, No. 10, Oct., p. 271
22. Spicer, *Transactions,* American Society of Mechanical Engineers, 1933, Vol. 55, I.S.-55-4
23. Cowan, Transactions, American Society for Metals, 1937, Vol. 25, Sept., p. 843
24. Guthrie and Wozasek, Transactions, American Society for Steel Treating, 1927, Vol. 12, Dec., p. 853
25. Wilbor and Comstock, Transactions, American Society for Steel Treating, 1930, Vol. 18, p. 533

DISCUSSION

Written Discussion: By John F. Wyzalek, chief metallurgist, Hyatt Bearings Division, General Motors Corp., Harrison, N. J.

The authors present a very interesting outline of past and present commercial practice of carburization of steel. Their extensive experience and thorough knowledge of the subject reflects itself throughout their splendid paper.

It is very interesting to read about their early attempts at gas carburizing. Our own, beginning about 1914, practically parallels theirs, except that experiments were conducted with manufactured or city gas.

Their statement relative to the quantity charged into a horizontal retort not to exceed two-thirds by volume proves interesting since our own experience, with possibly one exception, has brought about a similar conclusion, both from the standpoint of nicking and distortion of product as well as from the angle of uniformity of carburization. In the case of very small parts densely packed we found need to reduce the load to obtain desired results.

The emphasis placed by the authors upon uniformity of temperature as a very important and essential operating factor is fully justified. Our experience has borne this out, with both the batch and continuous type of gas carburizing equipment. It was found necessary to guard against forcing the heating of the product to be carburized since very ununiform case in depth and character would be obtained, even when using modern equipment of satisfactory design and construction. It is obvious that with compound carburizing, the material tends to act as a cushion and prevents excessive temperature variations of product. Perhaps due to this we are prone to conclude that gas carburizing is more sensitive from a temperature standpoint.

In conclusion may I add that the paper is a real contribution to the industry, offering operating data of real commercial value and presenting facts pertainng to the authors' own very successful plant operation, based on an extensive basis over considerable number of years.

Written Discussion: By Gordon T. Williams, metallurgist, Cleveland Tractor Co., Cleveland.

We are indebted to the authors for this excellent summary of current practice with a variety of equipment and media, as well as for the interesting history of their company's early work. Their description of their own operations is especially valuable.

The writer has experienced some difficulties with decarburization during direct quenching from a batch-type horizontal muffle gas carburizer which might be mentioned. The furnace holds six trays of work, and of course one tray at a time is taken to the quench tank, the load being then transferred to a basket and quenched. From 2½ to 5 minutes may elapse between removal of successive trays. Each time the door is opened air tunnels in, there being no vestibule or isolated discharge cell. If carburizing gas was left on, serious decarburization occurred on the trays remaining in the furnace. Flue (DX) gas was no better. "No gas on" gave some improvement in decarburization, but increased scaling. The best results were obtained when a shovelful of compound or charcoal was tossed in under the remaining trays as soon as the first tray was removed. This burned the incoming air to CO and CO_2 with-

out formation of water. By this means decarburization has been reduced to a trace on the second and third trays, none on the last three. When the load consists of parts later to be reheated, the trays are rapidly transferred to an insulated chamber for retarded cooling, the total transfer time being about 2 minutes, and decarburization does not occur.

The writer would like to suggest that there is a decided difference in tendency to scale, in air cooling from carburizing, between compound- and gas-carburized work. Compound seems to condition the surface to resist oxidation, while the surface from gas scales readily. No sound reason presents itself for this, but it has been observed many times.

Written Discussion: By J. A. Dow, engineer, Holcroft & Co., Detroit.

Messrs. Gable and Rowland are to be complimented on the clear and complete manner in which they have presented a tremendous amount of data on the commercial development of gas carburizing. Methods of operation have been carefully correlated to the results obtained.

The various gas mixtures in commercial use today are of course only necessary since a heavy soot deposit prevents feeding into a retort sufficient volume of undiluted hydrocarbon-rich gas to produce a uniform carburizing condition in all parts of an appreciable sized furnace. All diluting gases described by the authors have the common quality of being actively decarburizing in nature and complicate the chemical reactions which go on inside the carburizing chamber. The presence of these decarburizing agents necessitates a close regulation of flow conditions in the furnace to prevent nonuniform carburizing and have been responsible for a great deal of the apparently inexplainable variation in results which for several years placed gas carburizing in rather ill repute. We have never, however, found any decarburizing agents present in the gas coming from a successfully operating furnace, irrespective of the gaseous mixture fed to it.

We have recently put one of the largest continuous gas carburizing furnaces yet built into operation in Detroit using no generator to provide the necessary diluting gas. Part of the partially spent gas coming from the furnace muffle is enriched with natural gas and fed back into the furnace at a number of inlets.

The simplicity of this method in the elimination of the gas generator has an immediate appeal to any who have worked in the development of gas carburizing. The chief advantage gained is not, however, in the simplification of equipment but in the simplification of the chemistry inside the furnace muffle.

The process in commercial application is completely insensitive to flow conditions, making uniform results very simple to obtain. The character of case produced can be varied at will by the adjustment of one valve on the raw natural gas supply. There is apparently no need for any diffusion period since the carburizing activity of the gas can be held almost constant throughout the furnace length and can be regulated to produce a case of any desired carbon content with no apparent sacrifice in uniformity of results.

This method may be advantageously and simply applied to most small furnaces and we are sure no objection will be raised to its application by any furnace user.

Written Discussion: By P. C. Osterman, vice-president, American Gas Furnace Co., Elizabeth, N. J.

Mr. Gable and Mr. Rowland are to be commended for the excellence of their treatment of the subject of "Commercial Gas Carburizing." Their findings can well be accepted as authoritative since, unquestionably, they and their associates have supervised the successful gas carburization of a greater tonnage of quality steel parts than anyone else in the world.

On a subject of such vital interest differences of opinion are bound to occur; however, in weighing their remarks, we must give full consideration to the extended experience and broad background of the authors. They have covered this subject in a well nigh all-inclusive manner. They have also pictured in detail the procedure followed by their company so that one must be impressed with the measures provided to insure continuous duplication of quality carburizing.

The section under the heading "Control of the Process" could well serve as a summary of the subject. We must concur with the statements made in this section as to the importance of control of the temperature, the gas, the time, the steel and the human element.

It is believed that everyone will also agree with them that temperature is the most important variable which must be held under close control if consistent results are to be obtained. So long as the gas is sufficiently rich in available carbon, slight variations in it or in the rate of flow will not affect the final results. The other factors all have an important share in the results.

Too much emphasis cannot be placed on the statement of the authors that *"... Simplicity is a virtue .... not to be underrated."*

The possible variables are so numerous that it is highly to be desired that the number of these variables be held at a minimum and that additional variables be not introduced where they can possibly be avoided.

The general run of natural gas appears to be one of the most satisfactory carburizing agents which we have. Those who have it available are indeed fortunate. Where it is not available, the simpler the procedure which can be developed for providing a suitable carburizing gas the better. The same reasoning also, in our opinion, applies to the equipment in which this work is performed.

While gas carburizing is no longer a new subject, developments have been occurring at an increasingly rapid rate in the very recent past. That further developments will occur goes without saying. It would be difficult to predict the exact nature of these developments; but we may safely say that gas carburizing will continue to play an increasingly important part in heat treating. While the time required for gas carburizing has not been reduced to any appreciable extent in the past 30 years, I believe that we will shortly see a reduction in the time necessary for obtaining a given depth along with more complete control of the entire process. I am sure the authors will concur with me in this statement.

Undoubtedly Mr. Gable and Mr. Rowland can materially aid the further development of this process and we shall be interested in their future contributions.

Written Discussion: By L. A. Lanning, metallurgist, New Departure Division, General Motors Corp., Bristol, Conn.

The authors mention an installation using a propane-air mixture produced by means of a Selas mixer. I would like to ask as to whether or not in such an installation it is necessary to control the moisture content of the air used. My personal experience with this type installation has led to the belief that moisture is important.

I have not attempted to define moisture limits but have found that too thorough drying over activated alumina appears to retard the rate excessively. It also appears true that during periods of damp weather carburization proceeds at a faster rate than during dry weather. This has led to the belief that more uniform results might be obtained if the air were first dried and subsequently a definite amount of moisture introduced to the mixed gas. I wonder whether or not the authors have any data on this subject.

I note that the authors mention the extreme care necessary in dealing with gas carburizing and wish to add further emphasis to their statement. Gas carburizing for many purposes has great merit but is, as the authors state, a "ticklish operation" and requires close checking if it is to be successfully carried out.

Written Discussion: By Melville Lowe, metallurgist, Hevi Duty Electric Co., Milwaukee.

The introduction is interesting from a historical standpoint.

The early experiments at Timken Roller Bearing Co. are also of vast interest. The peach pits recall many other "Fairy Tales of Heat Treating." We find that it is very essential to carburize in a gas-tight container. Likewise, the description of the development of the American Gas Rotary Furnace is of interest.

The abstract of the paper states:

> "This paper reviews the development of gas carburizing from its beginning to the present day and discusses modern practice in this art. Commercial installations for gas carburizing are described in detail from the standpoint of the various gases used and information given regarding their operation. Variables affecting case depth and character of the case are discussed and recommendations made regarding commercial limitations in the control of these variables."

However, in fact, this paper has a tendency to overemphasize the operations of one company and is not a discussion of the art as a whole. I believe that so vast a subject should have been treated more generally.

The first line of the introduction states, "For the purposes of this paper alone, commercial gas carburizing is defined as those successful processes and practices in which the carburizing medium enters the retort in the gaseous state."

Electrically heated carburizing furnaces, for example, are not mentioned—only gas furnaces, with one mention made on the sixth page that "it can be fired with oil if desired." There are a large number of electrically heated gas carburizing furnaces in use today. These are in actual production, carburizing many thousands of pounds of steel, producing cases within close limit of depth and chemical composition. We have our own vertical carburizing furnaces of

which a large percentage are in operation using natural gas as a carburizing media. This natural gas is being used successfully without any preparation as drying, cracking, or diluting. We control the amount of soot on the work effectively and at will.

We use a fan in the cover, which produces turbulence within the retort and properly mixes the fresh gas with the spent gases in the retort, and recirculates this gas through the work where cracking occurs. This produces a uniform case. The composition and depth may be controlled at will.

Since it is a well known fact that electric heat is ideal from a uniformity standpoint, we satisfy the variable (a), page 732. Temperature. Within the retort of our vertical retort carburizing furnace we can easily maintain the variation stated by the authors.

(b) Gas

We can use natural gases, propane and butane, without any elaborate and complicated machine to prepare these gases. Rate of flow is easily controlled.

(c) Time

This divides itselt into parts:

First:—Heating-up time.

The electric vertical carburizer is powered high enough to heat its load in the minimum amount of time with a hot furnace—this is about two hours in most cases. Operating the fan during heating materially aids in uniform heating.

Second:—Carburizing time.

The carburizing media is not introduced until the work is at temperature. This carburization is started by introduction of the media.

Time is merely checked with a time piece.

(d) Steel:

Various steels carburize at different rates. This rate would be the same for the same steel at uniform temperatures and times.

(e) The Human Element:

We have found that it is very easy to instruct the average heat treater to become proficient in the use of our vertical carburizer furnace in a very short time. Place a man in charge of an electric furnace and he feels that he has a precision machine and treats it accordingly. We feel that we have reduced the human element to the minimum.

Natural gas does not exist everywhere and manufactured gas of 500-600 B.t.u. generally is a poor carburizer. For installation where gas is a question, we have developed our liquid Carbonol which is dripped into the hot retort, is gasified and passed through the charge. This is a very efficient carburizer.

The authors state on page 732 in reference to the temperature variation of plus or minus 10 degrees Fahr., "This small variation can only be maintained by continual checking and adjustment of the equipment, but it is felt that the effort will be repaid in results."

Throughout this paper mention is made of the fact that the gas-fired carburizer temperature is hard to control. Page 721, 16 lines from the bottom, we find this, "This small spread in work temperature (plus or minus 10 degrees Fahr.) is maintained only at the price of eternal vigilance." Therefore, this must be serious with their equipment.

A well designed electric furnace would be ideal in such a case.

In our electric vertical carburizer the basket can be fully loaded. It is not necessary to run partial heats. The advantages of this are many. The heating-up time is short. The charge, whether large or small pieces, is heated in the quickest manner by use of the fan.

The carburizing time is at a minimum. The fan materially shortens this. Soot is controlled at will without expensive auxiliary equipment. Work can be quenched from the carburizing temperature easily.

If it is desired to slow cool the charge, the retort and cover assembly may be transferred from the furnace to a cooling chamber. It is possible to cool at various rates. A second retort and cover can be charged into the hot furnace with its load and conserve the furnace heat.

The baskets with their charge may be transferred to a suitable cooling pit and a fresh set of baskets with a new charge placed in the retort and the cover put in place and operations continue.

In a recent installation, a furnace of 4600 cubic inches was charged with 400 pounds of bulky parts machined from Krupp steel. These were heated to carburizing temperature in one hour and thirty minutes, then carburized for twelve hours with natural gas. A case depth of 0.080 inch was obtained. This same charge was repeated with like results. This small electric vertical carburizing furnace replaces two large box gas-fired furnaces carburizing with compound. These gas-fired furnaces require 40 hours to produce a case of 0.072 inch.

A few comparisons of figures may be enlightening. The authors state, page 722, Table IV, small rotary 9000-cubic inch is 600 pounds. Our 4600-cubic inch furnace charged 400 pounds of a part similar to a bearing race. At this rate the 9000-cubic inch furnace would charge about 800 pounds. This is interesting from a cost viewpoint.

Our electric vertical carburizing furnace is at a disadvantage in localities where high B.t.u. gas is cheap and electricity high. However, the final costs are generally in favor of the electric vertical carburizer. Quality is also an important item in favor of electrically heated equipment.

Oral Discussion

H. W. McQuaid:[28] It seems to me that the subject of the intermittent flow method of gas carburizing has not been developed as fully as it might have been. That was the method originally developed by Guthrie, but without the use of any secondary gas, and primarily consists of determining, first, for a given steel, the amount and the time in which the work can remain in the gas-filled retort without new gas coming in, without decarburization of the surface. In other words, that is the time which governs the diffusion period, and the time the gas is on is determined by the time necessary to build up soot. In other words, if you have a certain given installation with a definite type of work and a definite gas to work with, you can carburize up to the point where soot would start to be objectionable, and then turn off the gas and let diffusion take place until surface loss in carbon starts.

One of the most successful installations that I know of uses a vertical-type

[28]Metallurgist, Republic Steel Corp., Cleveland.

retort in which the electric resistors for heating the work are inside the retort in the gas-filled chamber. They have determined in this installation that they can keep the gas on for approximately three minutes using straight and very rich natural gas, and by doing this they do not build up sufficient soot to cause an objectionable short circuiting of the resistors. The diffusion period required to give them the maximum carbon control in the case was twenty-seven minutes, so by combining the intermittent operation of three minutes gas on and twenty-seven minutes gas off they have been able to carburize nickel-molybdenum steel up to one-eighth of an inch, very uniformly and without any difficulty whatsoever as far as control of the carbon is concerned or the building up of soot on the electrical resistors. It seems to me that this installation indicates that this method has great possibilities. As Mr. Gable pointed out, the batch-type carburizing is about the only method at the present time whereby the straight rich gas, such as propane, butane, or natural gas, can be admitted directly to the retort, but by using the intermittent flow method it is possible to use almost any gas without particular difficulty, although the butane and the more unstable hydrocarbon gas, of course, might make the process difficult. With ordinary natural gas, or propane, the gas can be admitted directly into the furnace, and the intermittent flow method gives possibilities of control which seem to me to have been greatly neglected.

The previous discussor suggested that we dehydrate the gas completely and then add a specific amount of moisture. That has also been developed and that data published by Guthrie. I think it would pay us all to review the work of Guthrie and to give more attention to the intermittent flow method.

L. D. GABLE replies: The authors wish to thank the discussers of this paper for their kind comments. It is gratifying to know that our opinions regarding the control of gas carburizing are upheld by men of such experience in this field as you have heard discuss our paper today.

In regard to Mr. Williams' difficulties with decarburization we do not have such troubles because our entire furnace charges are quenched as a unit. We find it necessary, however, to keep the time interval between removal of the cover and actual quenching of the parts at an absolute minimum in order to avoid decarburization. The authors are aware of no better solution to Mr. Williams' particular problem than the one he himself has devised.

In reply to Mr. Lanning, our experience with propane and air mixtures has been limited to rather extensive experimental work. It is our opinion that satisfactory carburizing can be obtained without positive control of the moisture content but we agree that the control of the carburizing process in general is aided by close regulation of the moisture content of the entering gas. We also believe that the effect of moisture content is more pronounced in those processes which use diluted gases.

The process of gas carburizing described by Dow is being successfully used for producing the light cases. Since we have had no actual experience with this process we do not feel qualified to discuss its merits at this time, but we hope that in the very near future we will be able to familiarize ourselves with this interesting process so nicely described for us.

In respect to the intermittent surge methods, Mr. McQuaid, we would like to say that because of the shortness of time we gave only an abstract. We

did not give our entire paper. If Mr. McQuaid would be kind enough to read our paper in its entirety I am sure he will find our points regarding this subject in the paper.

Authors' Closure

The authors wish to thank Mr. Lowe for his description of the Carbonol process which we did not mention in the paper. As stated in the introduction we found it necessary to limit ourselves to a discussion of those processes included by our arbitrary definition of gas carburizing. We do not desire to infer that the Carbonol process is not of commercial importance. However, the authors are inclined to divide the broad field of carburizing on a basis of the physical state of the carburizing medium as it is ordinarily handled; i.e., solid, liquid, and gas. Any other basis for discussion is confusing because all commercial carburization is fundamentally gaseous in nature.

With regard to the balance of Mr. Lowe's discussion we feel that it is outside the scope of our paper. Methods of construction, heating, and mechanical operation of equipment were omitted because it was felt that this branch of the subject was worthy of separate treatment.

LIGHT CASES

By Vincent T. Malcolm

Abstract

This paper describes in brief the various mechanisms that require light cases. It points out the necessity of determining from the engineers standpoint the necessary qualities required in cased mechanisms, and describes methods for obtaining and reporting this information.

Variables that affect the measurement of hardness and the variation in instruments are discussed. Methods for measuring case depth are given and instruments for measuring the determination of brittleness and impact properties are described.

A general requirement for light case hardened steel is given together with the methods for producing light cases. Finally a description of results obtained with the Chapmanizing process on gears made from several steels is described.

As industry marches onward, new processes, new materials are constantly being developed and much attention is being directed to some of these processes, particularly the light case hardening of steel, because of its wide field of application and the publicity given.

The present discussion aims to designate the requirements necessary for the production of light cased parts from the viewpoint of the engineer.

What the engineer needs today is a comparatively cheap metal that will not have its physical properties greatly impaired by the service in which it is used. He needs a metal that will hold its surface under heavy sliding pressure, so as not to suffer from abrasion and exfoliation due to great velocity and pressure that may be used, and in order for a metal to have this resistance it must be stable under conditions outlined and be capable of resisting for the economic life of the equipment, of which it forms a part, stresses and wear to which it will be subjected in service.

The engineer is interested not so much in the metallurgical details of a given process as he is in new properties that can be

A paper presented as part of the Symposium on Carburizing presented at the Nineteenth Annual Convention of the American Society for Metals held in Atlantic City, October 18 to 22, 1937. The author, Vincent T. Malcolm, is Director of Research, The Chapman Valve Mfg. Co., Indian Orchard, Mass.

developed by a process in a metal, so that the latter becomes to him more useful or more economical than the metals or process he is now using; for there are constant demands made upon him to produce mechanisms to meet the ever increasing needs for stronger, faster, lighter and safer equipment, and this problem is therefore passed to the metallurgist, who must use all his skill and ingenuity to produce a product or process that will meet the demands of the engineer.

Extreme hardness and wear resistance combined with a low cost, easily machined steel, light in section, is the primary demand.

Light Cased Mechanisms

A few such articles requiring a light, hard wear resistant case may readily be mentioned as:—

Machine parts	Worms
Vacuum cleaner parts	Chucks and lathe centers
Bolts and nuts	Gages
Typewriter parts	Valve trim
Shackles	Chain links
Sewing machine parts	Ring and pinion gears
Textile machine parts	Spinning rings
Shafts	Cams and small gears
Timing gears	Refrigerator and electrical parts

and many other intricate parts that can be readily produced, but on which production is limited on account of costly regrinding after the hardening operation.

Hardness

From the engineering standpoint the first question asked is how hard is the case?

In order to attempt to answer this question intelligently it is first necessary to determine what is meant by hardness. The definition of hardness that will correspond to any physical value is a definition yet to be found.

Instruments such as Sclerescope, Rockwell, Brinell, Herbert Pendulum, Microcharacter, Vickers, Monotron, etc., used for hardness testing, all have scales obtained by different methods and anyone engaged in testing the hardness of metals will find that by use of these different methods they frequently show no definite rela-

tion to each other. From this we may assume there can be no correlation of the definition of hardness as applied to different methods of test for reason that considerable difference in hardness will be found when only the outside layers are measured or whether we measure deeply into the metal under test.

During the past several years we have attempted to study a correct definition of what hardness is, and after considerable re-

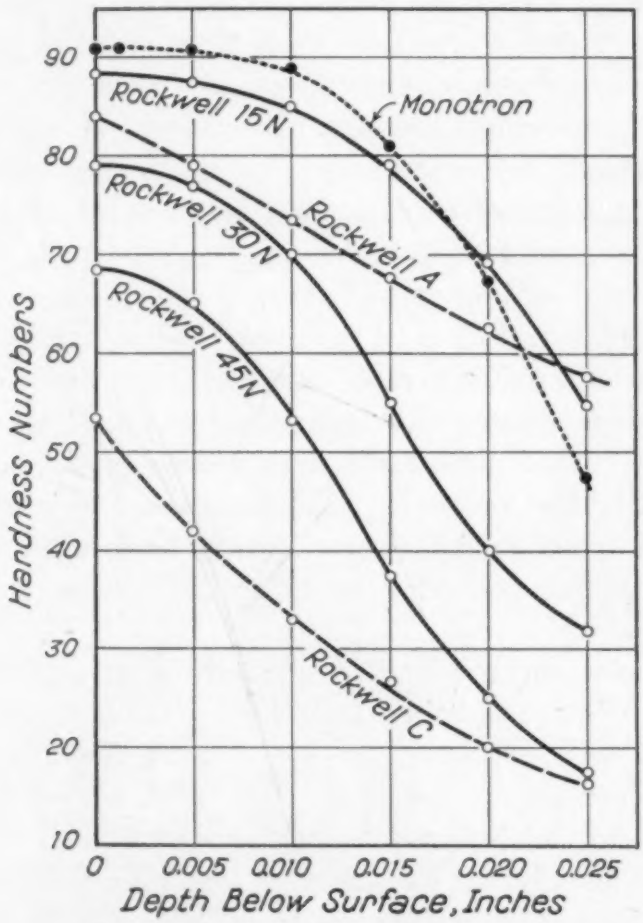

Fig. 1—Hardness as Measured by Several Types of Instruments.

search we believe that "by hardness is meant that property of mechanical resistance that is possessed by the outer layer of solid bodies." With this thought in mind it becomes necessary to measure extremely hard thin "cases" by certain specific instruments, especially designed for this class of work, such as the Herbert Pendulum, the Microcharacter, Vickers, Monotron, Hardometer or Superficial Rockwell, and by which the hardness so obtained may be

readily translated. Fig. 1 shows hardness as measured by several types of instruments.

Case Depth

This is another question usually asked in light cased work, and is one that is rather difficult to answer, because in most specimens there appears to be an outer hard case of one-third to one-half of the total depth of case; the remaining two-thirds or one-half is usually known as "transition case." This so-called transition zone must be the result of the case hardening process, because it is much harder than the core, and tapers off from extreme hard case to soft core in a normal manner. Therefore, if the specimen is polished and etched, while the entire case depth is shown there is no indication of its hardness at any point between the outer hard zone and the core, and where wear tolerance is considerable, hardness of this zone becomes an important factor.

In our opinion, the most accurate and satisfactory method, therefore, for determining true case depth hardness is obtained by the following procedure: A round bar is machined and ground to a uniformly exact diameter and then case hardened by any of the processes. After processing, the hardened bar is taper ground so that at one end the original case is untouched, while at the other end the grinding has tapered down to well under the maximum case possible.

The exposed surface is then marked off at regular intervals, each mark, for instance, being 0.005 inch greater than the previous one. At each of these several points the hardness is determined by one of the instruments previously mentioned, and successive readings from end to end indicate the hardness at various depths under the surface. The point on the specimen where hardness readings become constant, that is, where uniform hardness is obtained, is noted as the point of maximum penetration of the case, or case depth.

Wear Resistance

We probably know less about the mechanism of wear or how to test the wear resistance of metal than we do about other kinds of severe service conditions, and it is difficult to evaluate the wear factor in design and operation of equipment unless at least a relative wear resistance of the metal is known.

Very early in our work with light case hardened steel it be-

came evident that some relative wear test was necessary to determine the service possibilities of light cased steel. A wear testing machine was designed in our laboratory for such tests. This apparatus consists essentially of two carefully aligned specimen holders, the upper one being held in place by set screws. Through a speed reducing mechanism the lower holder describes a reciprocating motion, moving through a stroke of two inches, ninety times per minute. The specimens are thus caused to rub against each other in perfect contact, under a constant load. This load is transmitted to the upper holder through a special ended shaft, the weights being suspended below the machine. The specimens, which measure 1 x 2 x ¼ inch, are carefully ground to parallel faces before test and weighed after each 10,000 strokes to obtain loss. Through the use of this apparatus a wear factor has been established so that quantitative wear or wear resistance may be readily determined for various types of case and case depths.

The one property, therefore, that the engineer desires above all is service; or, in other words, resistance to wear in use for which the part is designed and performance of this function uninterrupted by shut-downs for repairs or replacements.

Brittleness and Impact

To the engineer the lack of brittleness and resistance to impact has an important bearing on the life of light case hardened metals. In our work a test for brittleness is made with a Brinell standard ball, a 3000-kilogram pressure, the ball being pushed into case, and indentation examined with magnifying glass at 10 diameters. If no cracking or checking is found, the case is considered to be ductile.

In the examination of light case hardened metal for resistance to impact, a repeated bending test is used to reproduce the stress conditions of service.

In the test, the bar is fixed in the test machine in such a position as to receive a blow on a reduced cross section, at the rate of 90 blows per minute with a hammer whose weight is five pounds and which hammer falls through a height of four inches. Between each blow the bar is rotated through 180 degrees and the test continued until failure occurs.

By carefully watching the test, crushing and spalling of the case may be readily determined.

General Requirements for Light Case Hardened Steel

1. *Parts*—Any piece in the machined condition, either forged, rolled, stamped or cast, should properly case.

2. *Steel*—Any carburizing grade of steel without special specifications.

3. *Hardness*—A Vickers reading of not less than 800.

4. *Case Depth*—From 0.005 to 0.040 inch as specified.

5. *Uniformity*—Depth of case and hardness to remain constant for types of steel treated.

6. *Growth or distortion*—Should be readily compensated by slight grinding or lapping operation in finishing the part.

7. *Finishing tolerance*—Should not be more than 0.003 inch on diameter.

8. *Straightening*—Parts that are necessarily thin or parts having different diameters or comparatively long pieces should straighten readily without cracking of case.

9. *Cleanliness*—Parts should be clean without great amount of washing.

Methods for Producing Light Cases

It is not the purpose of the writer to discuss the advantages of the various processes for obtaining light cases, and leaves this to the investigation of the user for obtaining the best process suited for the type of work at hand which will produce the best results with the least cost. However, it is not out of order to cite the following processes now generally in use for light case hardening of steel.

1. Sodium Cyanide Bath
2. Various types of activated baths
3. Rotary carburizing
4. Gas carburizing
5. Nitriding
6. Chapmanizing

The writer in closing this discussion would like to show, however, results that can be obtained in a well controlled process as Chapmanizing.

The tests were carried out on 4-inch 31-tooth gears made of several types of steel in order to obtain their characteristics in Chapmanizing.

The gears were made to a very close tolerance and were subject to exact measurements, to indicate their condition before and after treatment. The amount of shrinkage, growth, distortion or other physical changes were compared with the original and Table I gives the results of the findings. Test pieces of the same type of steel were treated along with gears to obtain the torsional and impact properties.

Gears 1-2-3 were S.A.E. 1025 Steel
Gears 4 were S.A.E. 2512 Steel
Gears 5-6 were S.A.E. 3250 Steel
Gears 7-8-9 were S.A.E. 3120 Steel

Hardness and Case Depth

		Monotron	Rockwell "C"	Case Depth Inches
Gears	1-2-3	890	58-59	0.022
Gears	4	860	62	0.024
Gears	5-6	910	63	0.020
Gears	7-8-9	920	63	0.022

Brinell Tests for Brittleness

A standard 10-millimeter ball was pressed into surface of test specimens with 3000-kilogram loading and each impression carefully examined with 10-diameter glass. In the examination no piece was found to be cracked or checked either at the surface or the bottom of the ball impression.

Table I

Paired gears	Average Backlash		Center distance		Runout		Diameter of hole	
	Before	After	Before	After	Before	After	Before	After
1-2	0.0015	0.0020	3.874	3.872	0.0015	0.0035	No. 1 1.000	.995
1-3	0.0010	0.0015	3.874	3.873	0.0015	0.0030	No. 2 1.000	.995
2-3	0.0050	0.0045	3.869	3.867	0.0020	0.0030	No. 3 1.000	.995
4-5	0.0020	None	3.874	3.880	0.0020	0.0025	No. 4 1.000	.998
4-6	None	None	3.879	3.887	0.0020	0.0020	No. 5 1.000	1.000
5-6	0.0060	0.0010	3.867	3.873	0.0020	0.0030	No. 6 1.000	1.000
7-8	0.0010	None	3.873	3.876	0.0030	0.0030	No. 7 1.000	1.000
7-9	None	None	3.877	3.878	0.0025	0.0025	No. 8 1.000	.999
8-9	0.0005	None	3.875	3.878	0.0030	0.0035	No. 9 1.000	.998

While the involute test data is complete both before and after Chapmanizing the gears, it is of such length and complication that it will not be presented in detail.

Test measurements were made on both right and left sides of the 31 teeth in each gear, before and after Chapmanizing, with the following results:

Gears 1-2-3 greatest change not over 0.0004 inch
Gears 4 greatest change not over 0.00015 inch
Gears 5-6 greatest change not over 0.0006 inch
Gears 7-8-9 greatest change not over 0.0002 inch

Gears 5-6 showed somewhat more distortion than any of the others. However, the greatest part of the distortion was concentrated in the teeth: that is, the run out and outside diameter concentricity with the hole were not appreciably affected; although the involute test showed that teeth were slightly elongated along a radial line.

Microscopic examination showed the case to be fine grain martensite free from needles and that it merged with the case without any sharp line of demarcation.

Torsional and Impact Properties

Separate test bars as previously outlined were tested in torsion and impact with the results shown in Table II.

TABLE II

Steel S.A.E.	Prop. Limit lb. per sq. in.	Yield Pt. lb. per sq. in.	Ult. Strength lb. per sq. in.	Gage Length inches	First Crack lb. per sq.in.	Total Twist degrees	Impact ft.-lbs.*
1025	33,400	41,300	74,600	7.68	61,800	133.0	10.0
2512	105,200	111,600	171,400	7.68	121,600	175.0	18.0
3250	111,000	119,100	167,500	7.68	92,550	71.0	6.0
3120	78,400	93,600	138,650	7.68	118,000	130.0	12.0

*—"V" notch.

Conclusion

In this paper we have only tried to present an outline of what is required in obtaining light case hardened steel properly processed, and no attempt has been made to describe processes or other details, because there are too many variations as to size of work, hardness, depth of case, time required, quenching, etc.; that only investigation and test of a process will give the final answer.

Oral Discussion

M. A. GROSSMANN:[29] In the measurement of the hardness of these very thin cases, what in your experience have you found to prefer as a measuring instrument?

V. T. MALCOLM: I believe the Vickers is probably the best measuring instrument for measuring case depth at the present time, and also depth hardness. There are a number of very satisfactory instruments among the instruments now put on the market which I did mention. I think I covered the list fairly well but I personally believe that the Vickers is the most satisfactory because you can get just the weights on the Vickers to give you a very light impression for case depth of just a few thousandths.

M. A. GROSSMANN: It seems to me that that is the point to be brought out because whenever there is an effect of the depth below the actual indentation there is liable to be an influence on the reading itself.

C. B. SADTLER:[30] I thought it might be interesting in connection with the discussion that has just been offered by the chairman and Mr. Malcolm to amplify a little further the relationship that exists between impression sizes of Vickers and the depth that is known to be effected. We have had occasion to carry the Vickers idea a little further than the usual range, namely down to 100 grams, which if spoken of in terms of the usual unit, kilogram, is 1/10, where the Vickers machine ordinarily is accurate down only to 5 kilograms. We have found that on Rockwell C equivalent of 60 hard material that 100-gram load on a Vickers point is influenced only by approximately 1000ths of an inch of metal of that hardness. For any other hardness and any load we have found that throughout the range from very soft to very hard material the depth that is affected is 1.4 times the diagonal of the impression. We usually say 1.5 to be a little safe.

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[30]Metallurgist, Barber-Colman Co., Rockford, Ill.

LIQUID BATH CARBURIZING

By B. B. Beckwith

Abstract

Liquid bath carburizing in the form of sodium cyanide for surface hardness has been used for a long time. Activated cyanide baths which increase the rate of penetration have become popular. This article shows what depth of case can be expected at different temperatures and different times. It also records the hardness obtained on four different scales of the Rockwell tester on the same piece.

Introduction

THE subject, "Carburizing," which is under discussion during this symposium, is one on which much has been said, more written and more yet to be investigated. This paper has to deal only with carburizing as accomplished in a liquid bath, its limitations, its difficulties and some typical applications.

The earliest liquid bath used for producing a hard surface was fused potassium cyanide. However, with the increasing use of this method of producing a hard surface, the change to sodium cyanide was but a short step and can be explained with one word—"cost." For a long time this was the only liquid case hardening bath used. The temperatures usually used varied from 1475 to 1550 degrees Fahr. and the time cycles 15 to 30 minutes. Consequently, the depth of case obtained was very thin and the piece was used as it came from the quench, cleaning being the only subsequent operation. If it were necessary for any reason to grind after hardening, then the cyanide bath was not used, the piece being pack carburized instead.

With the decrease in size of parts used in automotive construction, greater care had to be used by the metallurgist to maintain uniformity in the pieces used. This was especially true in those small pieces that required sufficient core for toughness and 0.010 to 0.015 inch case for wear resistance. With solid carburizer, packed in boxes and heated in furnaces, it was almost impossible to maintain necessary uniformity in quantity production of relatively small pieces.

A paper presented as part of the Symposium on Carburizing presented at the Nineteenth Annual Convention of the American Society for Metals held in Atlantic City, October 18 to 22, 1937. The author, B. B. Beckwith, is associated with the metallurgical department of the Chrysler Corporation, Detroit.

While sodium cyanide baths would give the necessary case depth required, if operated at proper temperatures and for a sufficient time, nevertheless, it was the activated cyanide compound baths which popularized the use of liquid baths for case depth up to 0.020 inch.

There are on the market several such compounds which have been proven commercially successful. Their activity in producing a hard case is based upon the reaction of the alkaline earth metals combined with the halogens or cyanogen with sodium cyanide and the iron of the part being treated. These compounds may be in one mixture or may be divided into two salts, one as the bath and the other as the additive compound. Another liquid bath not included in the above depends upon dissociated ammonia bubbling through the bath to hasten the reactions and produce the hard case.

It is not the purpose of the author to describe in this paper the various baths available, their chemical composition, reactions under heat or results of each individual bath. Nor will the type of case, carbon content, etc., and its effect on the hardness or wear be discussed. This paper will record only the average results obtained in all liquid baths which were used in this investigation. These results will include the hardness obtained and depth of case found at different times and temperatures.

Method of Test

The method of test used to collect data for this investigation was as follows:

3 steel analyses:	S.A.E. 1015
	S.A.E. 3115
	S.A.E. 4615
3 temperatures:	1500 degrees Fahr.
	1550 degrees Fahr.
	1600 degrees Fahr.
3 lengths of time:	30 minutes
	60 minutes
	90 minutes

The steels selected were the usual grades of carburized steels used in the automotive industry. They were approximately $\frac{9}{16}$-inch round bar stock, cold drawn and centerless ground in the bar. The temperatures selected were those commonly used for liquid baths.

The lengths of time selected were to give the effect of time on penetration. A furnace was selected of a pot capacity of about 100 pounds of compound. This amount of compound was used to start each bath tested, additions being made during the run to correspond with production practice. Each individual bath test was continuous. The three steels were all treated at each of the three temperatures listed for each of the three lengths of time. Six different baths

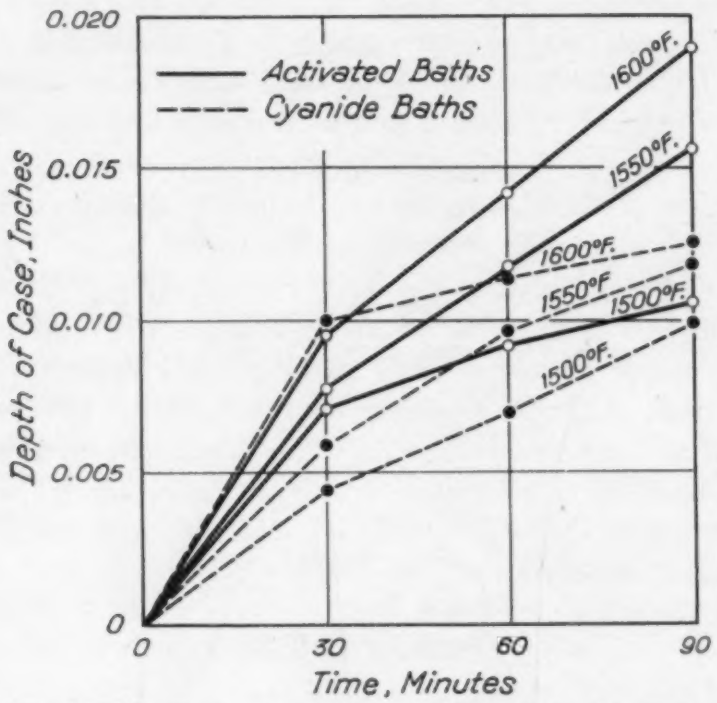

Fig. 1—Case Depths for Activated Baths and Cyanide Baths.

were used in this investigation. Each bath was heated to 1500 degrees Fahr. first, then about 30 pounds of scrap parts were added and all the samples of all steels to be tested at this temperature were suspended in the bath by means of wire. At the end of 30 minutes one-third of the samples were removed and quenched; the carbon steel in water and the alloy steel in oil. Likewise at the end of 60 minutes total time another third were removed and quenched and at 90 minutes the balance were quenched. The load of steel parts was then removed and the temperature of the bath raised to 1550 degrees Fahr., additions were made to maintain level and proper activity and above procedure repeated. The temperature was then raised to 1600 degrees Fahr. and above time cycles and quenching repeated. None of the quenched samples were drawn, being tested in the "as-quenched" condition.

Table I

Activated Bath—Rockwell Hardness—S.A.E. 1015 Steel

Temp. Degrees Fahr.	Time Minutes	15N. Aver.	15N. Max.	15N. Min.	30N. Aver.	30N. Max.	30N. Min.	45N. Aver.	45N. Max.	45N. Min.	C. Aver.	C. Max.	C. Min.
1500	30	92.1	95	90	76.1	80.0	70.5	58.3	66.5	46.0	40.8	48.5	30.5
1500	60	92.7	94.5	91.5	81.0	83.0	79.5	67.2	71.5	61.0	46.8	50.0	41.5
1500	90	92.3	95	90.5	80.8	83.0	80.5	68.6	71.0	66.5	47.2	53.0	41.0
1550	30	92.1	94.5	91.5	77.6	80.5	73.5	63.2	70.0	54.5	41.4	51.5	29.0
1550	60	93.2	95.5	92.5	81.9	84.0	80.5	71.8	73.5	70.5	52.2	56.5	48.5
1550	90	92.1	93.5	90.5	82.0	83.5	80.5	72.3	74.0	71.0	57.2	60.5	50.0
1600	30	92.8	95	91.5	80.3	82.0	77.5	65.1	70.5	57.0	47.8	53.5	39.0
1600	60	92.8	95	91.5	82.3	84	81.0	72.0	73.0	70.0	55.8	60.0	50.0
1600	90	93.0	95	92.5	82.6	84	81.5	72.5	75.0	69.5	60.9	63.5	59.5

Table II

Activated Bath—Rockwell Hardness—S.A.E. 4615 Steel

Temp. Degrees Fahr.	Time Minutes	15N. Aver.	15N. Max.	15N. Min.	30N. Aver.	30N. Max.	30N. Min.	45N. Aver.	45N. Max.	45N. Min.	C. Aver.	C. Max.	C. Min.
1500	30	91.1	94.5	89.5	71.2	74.0	68.5	49.8	51.5	46.0	27.1	31.0	26.0
1500	60	92.1	95.0	90.5	75.1	77.5	71.0	58.0	63.0	55.0	32.2	36.5	29.0
1500	90	92.0	94.0	92.5	74.7	80.0	75.5	62.1	66.0	59.5	36.9	47.5	28.5
1550	30	91.2	93.5	90.0	70.1	73.5	68.0	52.1	57.0	44.0	26.9	29.5	24.5
1550	60	91.2	94.5	88.5	77.2	83.5	74.5	61.7	67.5	57.0	37.2	44.0	30.5
1550	90	91.7	94.5	89.0	78.5	82.5	77.0	68.5	71.5	64.0	43.2	52.5	33.5
1600	30	91.7	95.0	90.5	73.6	76.0	71.5	54.7	58.0	51.0	29.2	34.5	23.5
1600	60	92.2	95.0	90.5	80.9	82.0	80.5	68.9	70.0	68.0	44.4	50.0	40.0
1600	90	91.8	95.0	89.5	80.5	83.0	77.5	69.7	71.5	67.5	53.4	59.0	48.0

Table III

Activated Bath—Rockwell Hardness—S.A.E. 3115 Steel

Temp. Degrees Fahr.	Time Minutes	15N. Aver.	15N. Max.	15N. Min.	30N. Aver.	30N. Max.	30N. Min.	45N. Aver.	45N. Max.	45N. Min.	C. Aver.	C. Max.	C. Min.
1500	30	88.8	91.5	86.5	67.5	69.0	64.5	46.8	51.5	44.0	26.0	29.0	23.0
1500	60	92.2	94.5	91.0	75.2	77.0	74.0	60.5	64.0	55.0	36.2	39.5	30.0
1500	90	90.8	94.0	87.5	77.7	80.0	74.0	63.7	65.0	63.0	41.2	44.5	39.0
1550	30	90.7	92.0	89.5	72.2	74.5	69.0	53.2	58.5	48.5	30.2	34.0	26.5
1550	60	91.0	93.0	90.0	77.2	81.0	75.0	63.8	67.5	60.5	38.8	45.0	32.5
1550	90	90.8	94.5	89.0	76.8	82.0	73.0	65.2	71.5	58.5	48.2	54.5	33.0
1600	30	91.8	94.0	90.5	76.2	77.5	75.0	60.5	63.5	58	35.2	37.5	32.0
1600	60	91.3	94.0	89.5	80.0	83.0	78.5	68.3	69.5	67.5	50.2	58.2	42.0
1600	90	91.2	95.0	88.5	78.8	83.0	76.0	64.8	71.0	66.5	54.5	57.5	49.0

Table IV

Rockwell Hardness—Cyanide

Temp. Degrees Fahr.	Time Minutes	1015 15N	1015 30N	1015 45N	1015 C.	4615 15N	4615 30N	4615 45N	4615 C.	3115 15N	3115 30N	3115 45N	3115 C.
1500	30	92.5	78.0	64.5	44.0	89.0	70.0	50.0	28.0	89.5	71.5	49.5	33.5
1500	60	91.5	79.0	66.0	50.0	91.5	75.5	59.5	36.0	89.5	75.5	58.0	36.5
1500	90	92.5	81.5	70.5	53.5	92.5	79.0	66.5	46.0	91.5	80.0	69.5	49.0
1550	30	90.0	71.0	53.5	34.0	91.5	68.0	58.0	37.0	88.0	65.5	49.0	30.0
1550	60	92.0	78.0	64.5	46.0	90.5	76.5	60.5	40.5	91.0	73.0	60.0	38.0
1550	90	92.5	79.0	69.0	53.5	90.5	78.0	63.5	36.5	91.5	78.5	64.5	41.0
1600	30	91.0	79.0	65.0	48.5	91.0	71.0	51.5	30.0	90.5	71.5	53.0	34.0
1600	60	91.5	80.0	70.0	52.0	90.5	73.5	51.0	31.0	91.5	78.0	64.0	43.0
1600	90	92.5	80.5	71.0	54.5	91.0	79.0	66.0	40.0	91.5	81.0	70.5	54.0

Table V
Cyanide Bath—Case Depths

(Inches)

Temperature Degrees Fahr.	Time Minutes	S.A.E. 1015	S.A.E. 4615	S.A.E. 3115	Average
1500	30	0.004	0.004	0.005	0.0043
1500	60	0.007	0.007	0.007	0.0070
1500	90	0.010	0.010	0.010	0.0100
1550	30	0.006	0.006	0.006	0.0060
1550	60	0.009	0.010	0.010	0.0097
1550	90	0.012	0.012	0.012	0.0120
1600	30	0.010	0.010	0.010	0.0100
1600	60	0.011	0.011	0.012	0.0113
1600	90	0.012	0.012	0.013	0.0123

Table VI
Activated Baths—Case Depths

(Inches)

Temp. Degrees Fahr.	Min-utes	S.A.E. 1015			S.A.E. 4615			S.A.E. 3115			Average
		Aver.	Max.	Min.	Aver.	Max.	Min.	Aver.	Max.	Min.	
1500	30	0.0074	0.008	0.006	0.0066	0.007	0.006	0.0072	0.008	0.007	0.0071
1500	60	0.0094	0.001	0.008	0.0092	0.010	0.009	0.0092	0.011	0.008	0.0093
1500	90	0.0118	0.012	0.011	0.0116	0.012	0.011	0.0120	0.013	0.011	0.0118
1550	30	0.0082	0.010	0.007	0.0082	0.009	0.007	0.0077	0.008	0.007	0.0080
1550	60	0.0120	0.014	0.011	0.0120	0.014	0.011	0.0120	0.013	0.011	0.0120
1550	90	0.0152	0.017	0.014	0.0154	0.018	0.014	0.0162	0.018	0.015	0.0156
1600	30	0.0098	0.008	0.011	0.0088	0.011	0.007	0.0092	0.010	0.008	0.0093
1600	60	0.0140	0.015	0.013	0.0142	0.015	0.013	0.0145	0.015	0.014	0.0142
1600	90	0.0185	0.020	0.018	0.0184	0.019	0.017	0.0182	0.019	0.016	0.0184

The hardnesses recorded were obtained on a Rockwell Superficial and a Rockwell Standard testing machine. The results recorded in Table I are the average results for all baths for the same time and temperature. As the cyanide bath is the most familiar, it is listed separately in Table V. The depths of case are also average for all baths, as listed in Table VI. The curve (Fig. 1) for case depth is the average for all steels as well as all baths.

Analysis of Steel

S.A.E.	C	Mn	S	P	Si	Cr	Ni	Mo
1015	0.18	0.46	0.015	0.023	0.23	nil		
3115	0.17		0.020	0.025	0.16	0.63	1.25	nil
4615	0.17		0.020	0.038	0.19		1.78	0.25

Grain Size (McQuaid-Ehn)

S.A.E. 1015—5-6 with same larger than No. 1
S.A.E. 3115—5-7 occasional 3
S.A.E. 4615— 8

Analysis of sodium cyanide bath.

Cn calculated as NaCn.

at the start of 1500 degrees Fahr. run	21.7 per cent
in the middle of 1550 degrees Fahr. run	23.9 per cent
at the end of 1600 degrees Fahr. run	23.3 per cent

10 pounds of 96-98 NaCn were added at the start of both the 1550 and 1600 degrees Fahr. runs.

Method of Measuring Case Depths

To measure the case depths of the samples, the pieces were cut with a rubber wheel, the end ground smooth and polished. They were etched with a 5 per cent solution of nitric acid in alcohol and the total darkened area measured with a Brinell microscope.

Discussion of Hardness

Examination of the hardness results from the use of 15N Rockwell Superficial shows that the skin hardness values obtained on the outside surface were approximately the same for the various baths within limits of experimental error, and regardless of the time or temperature. As the load is increased, by using a different hardness scale variations can be found between the same time and different temperatures and different times and same temperature. These variations are to be expected. The results on the different steels at different times and different temperatures are included in the hope that they will be found interesting to many readers. Likewise, as the load of the hardness tester is increased, the effect of the core hardness immediately under the case enters into the ultimate reading. The thickness of the case, or what might be called the load-carrying capacity, also effects the hardness obtained.

Depth of Case

In examining the case depth obtained by the different baths it was found that very little variation was apparent between the different steels when run together at the same temperature for the same time and, therefore, the average results were plotted in Fig. 1. Straight sodium cyanide is slower in producing case depths than the activated baths. The penetration is most rapid during the first few minutes.

Suppose the service requirements of a part are such that a minimum of 0.012 inch case was desirable and the section such that carburizing in solid carburizer was not feasible. Liquid bath carburizer was selected as a means of obtaining this depth of case. Examination of the curves showed that 0.012 inch can be obtained in 60 minutes at 1550 degrees Fahr. or 46 minutes at 1600 degrees Fahr. in the activated baths or 74 minutes at 1600 degrees Fahr. in sodium cyanide. Inasmuch as cyanide breaks down rapidly and is lost by volatilization at 1600 degrees Fahr. and as well as the

length of time required, the activated baths are to be preferred. Experience over several years' operation of activated baths has shown that 1550 degrees Fahr. is the most satisfactory temperature for carburizing in activated baths. It must be remembered that the depths recorded are average results. The depths would vary with type of furnace, ratio of weight of work to weight of salt, accuracy of temperature control and other variables inherent in liquid bath operations.

Advantages of a Liquid Bath

The very nature of a liquid bath is such that the air is excluded during the heating cycle, thus preventing oxidation. Likewise, during the brief interval in the transfer from the bath to the quenching medium, a film of compound adheres to the piece preventing oxidation. The quenching medium or wash tank removes this film and the surface is left as clean or cleaner than before hardening. In comparison with solid carburizers, the temperature usually used (1550 degrees Fahr.) being much lower minimizes the warpage or distortion. For these reasons many parts can be finish machined and ground prior to hardening, and full advantage taken of the extreme skin hardness. Heat transfer in a liquid bath is fast, and the work in a salt bath comes up to heat rapidly, and the time at heat can be carefully controlled, and this time determines the depth of case obtained. The parts are direct quenched, thus saving a reheating.

Typical Applications

Liquid bath carburizing is especially suited for small stampings or parts turned from bars which have thin sections, and which must have a thin case. Examples of these are speedometer gears and pinions, thrust washers, clutch throwout forks, bushings, throttle control parts, and small shafts. It can be used to advantage on parts where the loads are relatively light, and the wear of a few thousandths would make it inoperative or ineffective.

Precautions

Parts carburized in a liquid bath usually require a thorough washing or neutralizing dip after removal from the quench tank to insure against rusting. It must be remembered while operating a bath that both the work and the heat are removing the active car-

burizing agent, therefore, if the "drag-out" is insufficient to require addition of the base compound from time to time, to maintain level, then it is necessary to "bail out" some compound and add new. The amount and frequency must be determined on each individual furnace and part carburized. Each metallurgist must determine for himself, from the characteristics of the bath, which one will fit into his operations and equipment to the best advantage, if he desires to use activated liquid bath carburizing.

Acknowledgment

The author wishes to thank the members of the staffs of the various activated salt manufacturers who have so generously assisted him with their help and advice, Mr. Dexter of the Chicago Screw Co., the following members of the Chrysler Engineering Metallurgical staff who contributed their help: A. J. Gerhardt, A. B. Schump, and J. D. Loveley, and also the Chrysler Corp. for permission to publish the data.

DISCUSSION

Written Discussion: By V. E. Hillman, director of research, Crompton & Knowles Loom Works, Worcester, Mass.

The paper is indeed informative. It shows conclusively wherein the various liquid compounds excel over the straight sodium cyanide bath. Briefly, the depth of case is more pronounced after the longer periods of immersion.

There is one feature which the writer wishes to point out, however, when using these compounds. On work which requires only partial immersion proprietary compounds have a tendency to form a deposit at the line of immersion which is exceedingly difficult to remove with the ordinary cleaning solutions. The difficulty is not experienced in the sodium cyanide bath.

Written Discussion: By R. H. Webber, general foreman of heat treating, Cadillac Motor Car Co., Detroit.

As Mr. Beckwith states in his opening paragraph, the subject, "Carburizing," has been in the past very well covered and much more should be investigated in the future. The activated liquid carburizing bath method, however, has not had this prominence and as it has promises of a great future, anything that may be contributed to this particular phase of carburizing will help to develop its more universal use. Mr. Beckwith is deserving of much credit for presenting the facts in his paper and as a whole I can heartily agree with most of his statements.

The writer of this discussion desires to bring out some of the facts, perhaps not covered previously. It becomes more difficult to obtain a case depth of 0.030 inch with a Rockwell hardness of C 60. It is our experience to obtain

this in an activated liquid bath with production methods. Using a 24-inch diameter by 18-inch depth pot with about 300 pounds of compound, a constant load of 40 pieces of 2¼-inch diameter shafts of S.A.E. 4620 steel is immersed in the bath. This load makes a total of 200 pounds or 66 per cent of compound weight, which is considerably more than previously attempted. A distortion change of not more than three-tenths of one thousandths of an inch must also be maintained. An hour and a half is required to bring the bath "to heat" at 1600 degrees Fahr. and an average of three and a half hours is required at heat. An individual drastic quench in oil results in the previously mentioned Rockwell test of C 60 and a 0.030-inch case depth.

To obtain these results consistently, many factors which enter the cycle to cause disturbances, or loss of the chemical balance between base and catalyst, must be considered. A pot of pressed steel, cast steel, or an alloy with not more than 35 per cent nickel is preferred. If the pot is brought to heat too rapidly, there is a tendency for the compound to break down in the lower section of the pot. A proper graphitic cover, coat, or blanket is preferred when desiring case depths of 0.030-inch, and it has also been found to be very essential in the lighter case depths. It is common practice to have some drag-out of compound as it adheres to the parts on quenching and new materials must be added to maintain the bath level. This drag-out in baths, intended for heavy case depths, does not represent sufficient replacement to maintain the proper chemical equilibrium; but it becomes necessary to bail out enough compound from the bath to allow for proper additions. These additions should preferably be made at the beginning of each load. Due to the adherence of the compound to the stock during quenching operations, it sometimes becomes a problem to economically remove, but a proper compound can readily be removed in hot water if time is allowed to soak it off.

While we have not added materially to the technical phase of the subject, perhaps these few practical additions may be an aid to the new user, for the writer has done nothing more than to record a few personal observations made at close hand for the information and guidance of those who can use it.

Written Discussion: By Haig Solakian, A. F. Holden Company, New Haven, Conn.

Mr. Beckwith's paper presents, rather briefly but clearly, some of the fundamental facts involved in the use of liquid carburizing baths. The results of carefully carried out tests, as tabulated in the paper, answer a great many questions which are often left to guesswork. The rate of case penetration on various types of S.A.E. steels, the depth of case for various times and temperatures, these are questions of everyday practical importance and the paper answers them quite adequately.

Mr. Beckwith considers the most effective factors that control the depth of case, such as time and temperature. To these may be added another one, namely, the size of piece to be carburized. At the same time and temperature a large piece does not carburize to the same depth as a small one. This particularly applies to parts of round cross section. In figuring the time and temperature requirement to obtain a certain case it is worth while to bear this point in mind.

In measuring the depth of case Mr. Beckwith employs a method of polishing and etching. It seems that this is a rather lengthy and time-consuming

process. It has been our experience that using a telescope magnifier to about 10 x 20 diameter with an objective graduated to thousandths of an inch and taking readings directly on fractured surfaces gives quick and accurate results. Larger pieces difficult to break may be notched before treating. This fracture method has been used on pieces as large as two inches in diameter.

Mr. Beckwith states several interesting advantages of a liquid bath. To these we may add a few that are important, especially for liquid carburizing baths.

The characteristic which contributes to the success of a liquid carburizing bath very effectively is its controlled rate of decomposition. It is due to a great extent to this fact that the bath carburizes in a shorter time and, further, it is also for this reason that tons of work can be put through continuously for days with a uniform case and hardness. With an ordinary sodium cyanide bath the rate of breakdown or oxidation is very rapid. Conservatively speaking this rate is about three times as fast as with a liquid carburizer. Usually there is also a low limit for a cyanide bath necessitating the presence of approximately 15 per cent sodium cyanide below which the bath ceases to function properly. With a liquid carburizer the percentage of cyanide may drop slowly, of course, and yet the casing action continues satisfactorily irrespective of the percentage of cyanide. We have noted on various occasions that a liquid carburizing bath with 3 per cent cyanide functioned steadily in production.

The precautions sounded by Mr. Beckwith for a proper balance of drag-out and new additions are very timely and important. The amount of effective carburizing agent in any bath is fixed. If we express this fixed amount of agent as "fixed energy" we may say that although the rate of breakdown or consumption of this energy is slow, if more is taken out than is put in there will come a time when the energy will be entirely consumed and the bath will eventually cease to carburize.

These precautions would not be complete without a warning regarding the contact of nitrates with liquid carburizers. These latter contain various percentages of cyanide and any tempering bath of a nitrate nature should be kept away from these carburizers.

Before concluding we would like to enter a plea for the judicious consideration of the depth of case that is actually necessary for any part that needs to be carburized. Very often we have seen parts where 0.030-inch case was specified while 0.015-inch would have been amply sufficient. An exceedingly deep case does not necessarily render the part better. An unnecessarily heavy case not only costs more to put on, but also cost more to grind off and finish to size later on. Our plea would be to consider the service requirements of the part and aim for a case that would meet these requirements.

This investigation was carried out with a frank open-mindedness and presented with judicious fairness, for which Mr. Beckwith is to be complimented.

Written Discussion: By H. E. Martin, E. F. Houghton & Co., Detroit.

The writer has laid the foundation for a great deal of necessary constructive work on the salt baths. The information contained in the paper is invaluable for the engineer, metallurgist and operating personnel as a guide for the proper selection of a medium and procedure to follow to arrive at the desired hardness and case depth.

There are some irregularities in the hardness readings and case depths, but I believe that if a greater number of samples had been checked, the figures would show a greater consistency. As an example the average case depth obtained in cyanide at 1600 degrees Fahr. for 30 minutes is somewhat higher than would be expected.

A feature of some activated baths which was not mentioned in the paper is the carbon covering on the bath which reduces radiation from the bath and as a consequence reduces the heating time of the charge.

At a future date I am hoping Mr. Beckwith can delve further into the activated baths and obtain data on microstructures; also carbon and nitrogen concentrations in the case and reactions occurring in the bath.

Oral Discussion

J. F. WYZALEK: Not having the opportunity of reading Mr. Beckwith's very splendid and capable paper I am not in a position to know whether or not he has covered the matter of microstructure. I feel that that does play quite an important part in the question of wear resistance and fatigue, and I hope I am not out of order in suggesting that perhaps in his written reply he might consider including some photomicrographs of the various types of structures produced by methods described in his paper.

SAM TOUR:[21] Mr. Beckwith is to be complimented on having done a valuable piece of work in preparing this paper. I do not care in any way to detract from the value of that work. However, it might be well to bring out a number of points which seem to me very important in connection with any such study of cyanide versus activated baths. I hold no brief for cyanide baths. However, I do not believe this paper is a fair comparison or a fair test of cyanide versus liquid bath carburizing.

The author states that cyanide baths as a general rule are used at temperatures from 1475 to 1550 degrees Fahr. As a matter of fact, I think those temperatures are very high. Cyanide baths have always been known to break down rapidly and not to be economical for use at such temperatures as 1550 degrees Fahr. Normally they are used at 1350 to 1500 degrees Fahr. In other words, 1500 degrees Fahr. should be the maximum temperature, whereas that was the minimum temperature used by Mr. Beckwith.

It has been common knowledge for many years that cyanide baths break down very rapidly at high temperatures and also it has been common knowledge that certain minimum amounts of cyanide must be present if proper case hardening is to be accomplished.

Mr. Beckwith gives some analyses of cyanide content. Unfortunately he does not give a complete set of analyses. It is necessary, therefore, to estimate the actual cyanide content which was in his bath for the different treatments. These estimates as based on his figures show that the 1500-degree Fahr. test started out with less than 22 per cent cyanide and finished with about 16 per cent cyanide. In other words, sufficient cyanide was not present in the 1500-degree Fahr. bath and the curve shows a low case. At 1550 degrees Fahr. the test started out with 26 per cent cyanide and finished with 20 per cent. This bath was really

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[21] Vice-President, Lucius Pitkin, Inc., New York City.

low in cyanide but higher than the 1500-degree Fahr. bath. The curve naturally shows a higher case. At 1600 degrees Fahr. the test started out with about 33 per cent cyanide and finished at 23 per cent. The result was a higher case. The three cyanide tests within themselves are not properly comparable because a uniform cyanide content was not maintained.

Some question may well be raised on the method of measuring case depths. It has been known for many years that cyanide has both a carburizing and a nitriding effect. The higher the temperature the lower is the nitriding effect and the more effective is the carburizing. The particular method used by Mr. Beckwith for determining the case depth was not one that would take into account and evaluate the nitriding effect.

The method of measuring case depth by simply polishing a surface and etching with 5 per cent nitric acid in alcohol or water is hardly a proper method for comparing a cyanide case with a carbon case.

Cyaniding should be considered as having its particular field, a field limited to temperatures of 1350 or 1400, 1450 degrees Fahr. possibly, and where nitriding as well as carburizing is permissible or desired.

Where the nitriding effect is objectionable or where a high carbon or high depth case is desired the activated baths are indicated. There is a definite field, in my opinion, for each type of bath.

May I discuss something not directly connected with this paper but connected with the carburizing symposium as a whole? Is it possible to determine the efficiency of carburizing by studies of case depths? In all this carburizing work, it should be kept clearly in mind that there are two different things going on at one time. First, carbon is being supplied to the surface. Here the carburizing reactions themselves must be considered. Second, having supplied that carbon to the surface, a separate and distinct reaction comes into play—the diffusion of carbon within the steel. Only confusion results from attempts to compare two different carburizing materials by simple studies of case depths. Case depths are a function of time, temperature and concentration gradients of carbon in the steel. Only the maximum carbon near the surface has to do with the carburizer itself. Carburizing stops when the carbon supplied to the surface has yielded a surface carbon content peculiar to the carburizing agent being used. This is the carburizing reaction. Case depths have to do with diffusion rates within the steel, are peculiar to the steel and for a given temperature and outer surface carbon content are independent of the source of carbon or carburizing reaction.

Numerous published papers, and some given in this symposium, deal with the carbon content and depth of cases with respect to certain carburizers and temperatures of carburizing. There seems to be an opinion that a higher carbon case is obtained with a lower carburizing temperature. This is not true. With any given solid carburizer, the higher the carburizing temperature the higher is the carbon in the case, until diffusion within the steel takes the carbon away from the surface faster than it is supplied. To study carburizing as such, it is necessary to use samples of such small cross section that they will carburize all the way through and diffuion will not interfere with carburizing. Doing just that shows that the carbon resulting from carburizing with a given carburizer increases with the temperature of carburizing. Studies of the carburizing re-

action itself should be made on samples in the form of strip 0.01-inch thick or wire 0.020-inch in diameter. Diffusion of carbon in steel is a separate and distinct phenomenon and should not be allowed to confuse the study of the phenomenon of carburizing.

J. I. MEDOFF:[32] Just what, if any, are the effects on the hardening of the work and sludging of the oil bath, of the salt or cyanide film on the work, when oil quenching directly from the pot?

Author's Written Reply

The number of discussions received on this paper indicates the great amount of interest in the subject. The writer wishes to thank those who discussed the paper and added information and value to it.

With reference to Mr. Hillman's comment on the difficulty of removing the deposit on partial immersion work, I wish to state that his point is well taken. However, our practice has been to finish clean the majority of heat treated parts with a light sand blast. With this practice, the difficulty has been simplified.

Mr. Webber's comment, relative to partial carburizing a shaft to 0.030-inch, is very interesting as it is a concrete example of a unique application of liquid bath carburizing. The case is deeper than usually found commercially economical, but the mechanical limitations are such that other methods of obtaining the case would be more difficult. There are many such special applications that can be found if a study of the individual parts is made.

Dr. Solakian adds considerable information on application and operation of liquid baths. It was realized that size of piece carburized would affect the depth of case, and, therefore, this variable was omitted intentionally. Perhaps in reading the method of measuring case depth, he misinterpreted the method of polishing. It is a rough grind to take off enough material to get through the case to the core followed by a polish with a felt wheel on which 120 abrasive is glued. This method has been found to be just as rapid on a great number of differently shaped pieces as the fracture method.

Mr. Martin mentions the action of a carbon covering on the bath. Some baths function better with a cover, other baths do not require it. As for structure and constitution of the case, it was not the purpose or intention of this paper to discuss this phase of liquid bath carburizing.

Mr. Tour, as far as the temperature which was used, 1450 to 1475 to 1550, that is among the automotive industry a very common temperature for cyaniding. Lower temperatures are used but not to any great extent in the automotive industry around Detroit, at least. As far as the cyanide content of the cyanide baths, it was the intention of this paper to present a production result which could be repeated right in the heat treat on an hour-after-hour, day-after-day basis and not a laboratory controlled practice. In that case the production procedure was stopped, the bath was taken as is, cyanide additions made according to standard practice and the samples run at the various temperatures indicated. In that way, naturally, the cyanide content would be lower than would be made

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[32]Worthington Pump & Machinery Corporation, Harrison, N. J.

had we started out with a fresh bath and maintained on a laboratory test procedure.

In checking case depth, there again it was entirely a production practice and not a laboratory method, although it was done by our own laboratory and our own laboratory men.

THE PROCESS OF DRY CYANIDING

By R. J. Cowan and J. T. Bryce

Abstract

The processes of continuous nitriding and continuous gas carburizing have been combined to produce a case in many respects similar to a cyanide case. The results obtained with different steels by various modifications of the process are presented as a series of micrographs and charts. The development of the process is briefly described and there is some discussion of the results obtained.

WHEN dealing with the case hardening of metals, the investigator finds himself in a field of very ancient lineage. The means for adding carbon to the surface of a metal is one of the oldest of the metallurgical arts. It is an extremely interesting matter to follow along through the years the many theories that have been propounded which have given rise to varying schools of thought. Each of these has had its own high priest, apostles, disciples and martyrs. It is a significant fact that the more important of these theories, which at times have been discredited and overthrown, have from time to time been resurrected and clothed in a new form ready again to carry on another warfare in the cause of truth. It would seem to be fitting and proper at the beginning of any new entry into this field to pay our respects to the early investigators whose favorite ideas may be found to have been incorporated in the things we are doing today. If it were possible to survey all the voluminous literature on this subject, we would probably find that our best suggestions are only a reincarnation of ancient thought in modern garb. No attempt will be made to review this early literature in its bearing on the matter being discussed. It is only necessary to survey the field in a general way in order to bring to mind some of the outstanding facts.

The use of charcoal for adding carbon to iron is one of the earliest practices. It was soon discovered that the effectiveness of

A paper presented before the Nineteenth Annual Convention of the Society held in Atlantic City, October 18 to 22, 1937. Of the authors, R. J. Cowan and J. T. Bryce are metallurgical engineer and metallurgist, respectively, Surface Combustion Corp., Toledo, Ohio. Manuscript received July 24, 1937.

the carbon could be very much increased by the addition of various substances to charcoal. This led through a series of elaborate experimentations which covered the use of a great variety of mineral and organic materials, many of which were compounded in a curious manner. Arising gradually from this experimental work, there has been developed the efficient carburizing materials with which we are familiar today. It was frequently observed in this early work that the presence of nitrogenous materials along with the carbonaceous materials not only affected the case hardening rate but also the character of case produced by the treatment. Such materials as charred leather, horn scrapings and fish scales, were very widely used.

When the process of carburization by means of gas was developed, it was found that a case could be produced upon a metal either by the use of carbon monoxide or of a hydrocarbon gas,—the character of this case being distinctive in each instance. It was also observed that the addition of a nitrogenous gas or vapor had an effect upon the case produced which was similar to the effect of nitrogen upon the charcoal case. The use of ammonia for this purpose along with a carburizing gas is of very early origin. The present paper deals specifically with the adaptation of the use of ammonia to the process of continuous gas carburizing in such a way as to produce a case of controlled nitride and carbide content which operation we designate as the process of gaseous or dry cyaniding.

The results described in this paper are taken from a vast series of tests extending over a period of several years. It would be impossible to describe all these tests in detail. An attempt is made, however, to point out the more important results that have been obtained and to indicate what the possibilities are for this method of case hardening.

First Series

In the first series as presented, a comparison is made between carburizing and dry cyaniding at the same temperature. A variety of standard S.A.E. steels have been used, and, for comparison, these have been given three separate treatments.

First, a carburizing treatment consisting of a hold for one and one-half hours at a temperature of 1450 degrees Fahr. in an atmosphere composed of 40 per cent natural gas and 60 per cent DX gas.

Second, a similar lot of test specimens held for the same time at

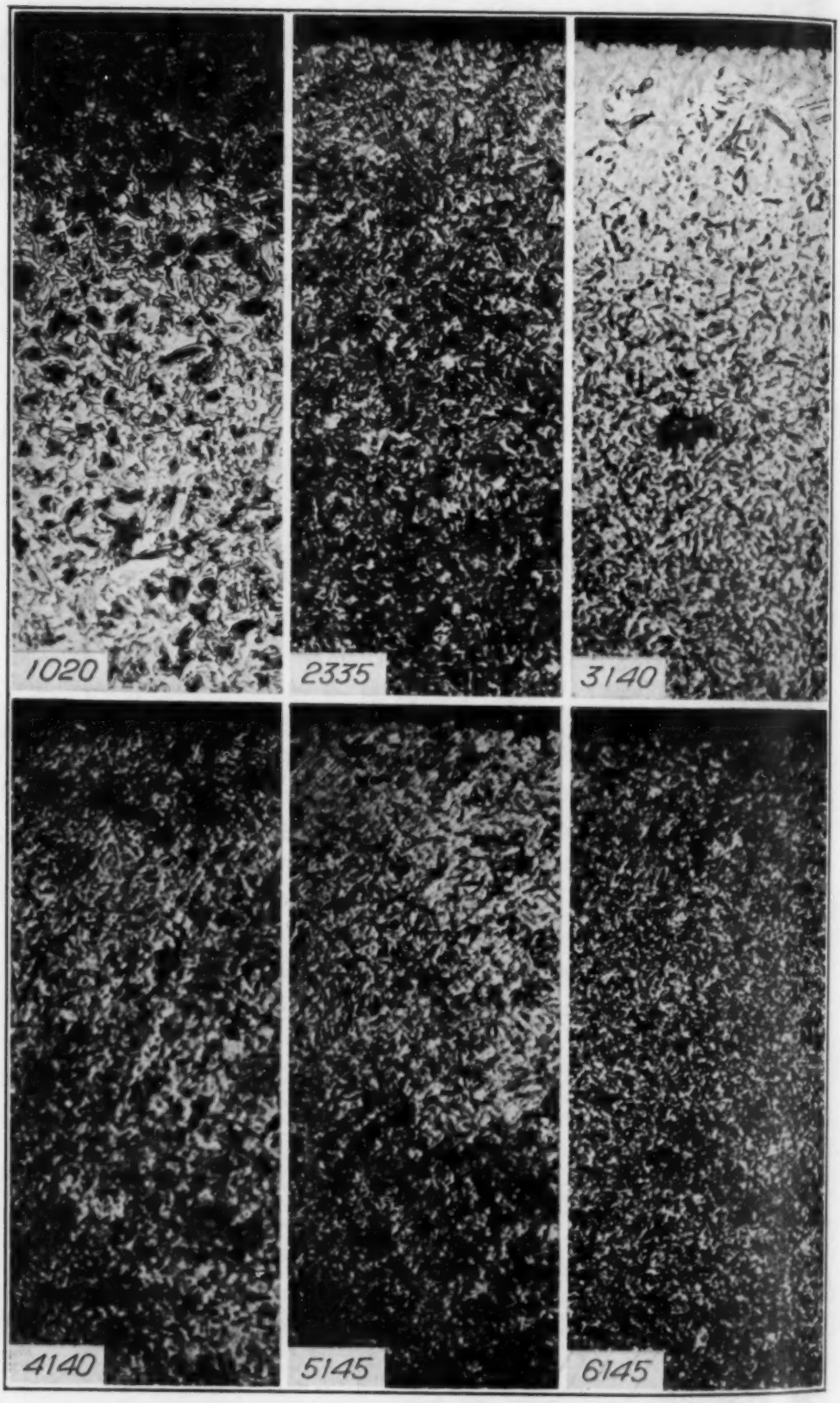

Fig. 1—Carburized S.A.E. Steels. Natural Gas Plus Dx Gas. Temperature 1450 Degrees Fahr. Oil-Quenched.

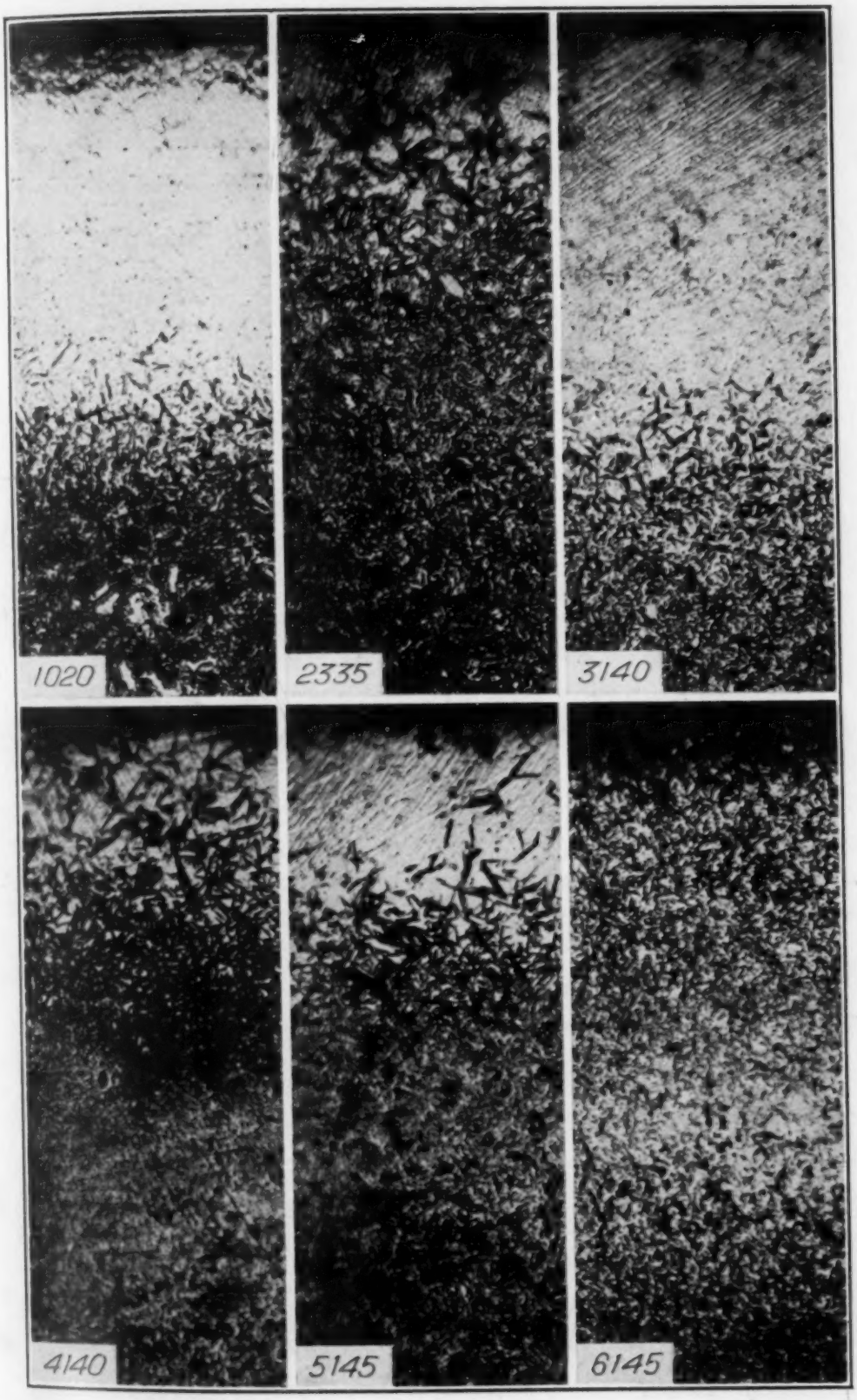

Fig. 2—Carburized S.A.E. Steels. Natural Gas Plus Ammonia. Temperature 1450 Degrees Fahr. Oil-Quenched.

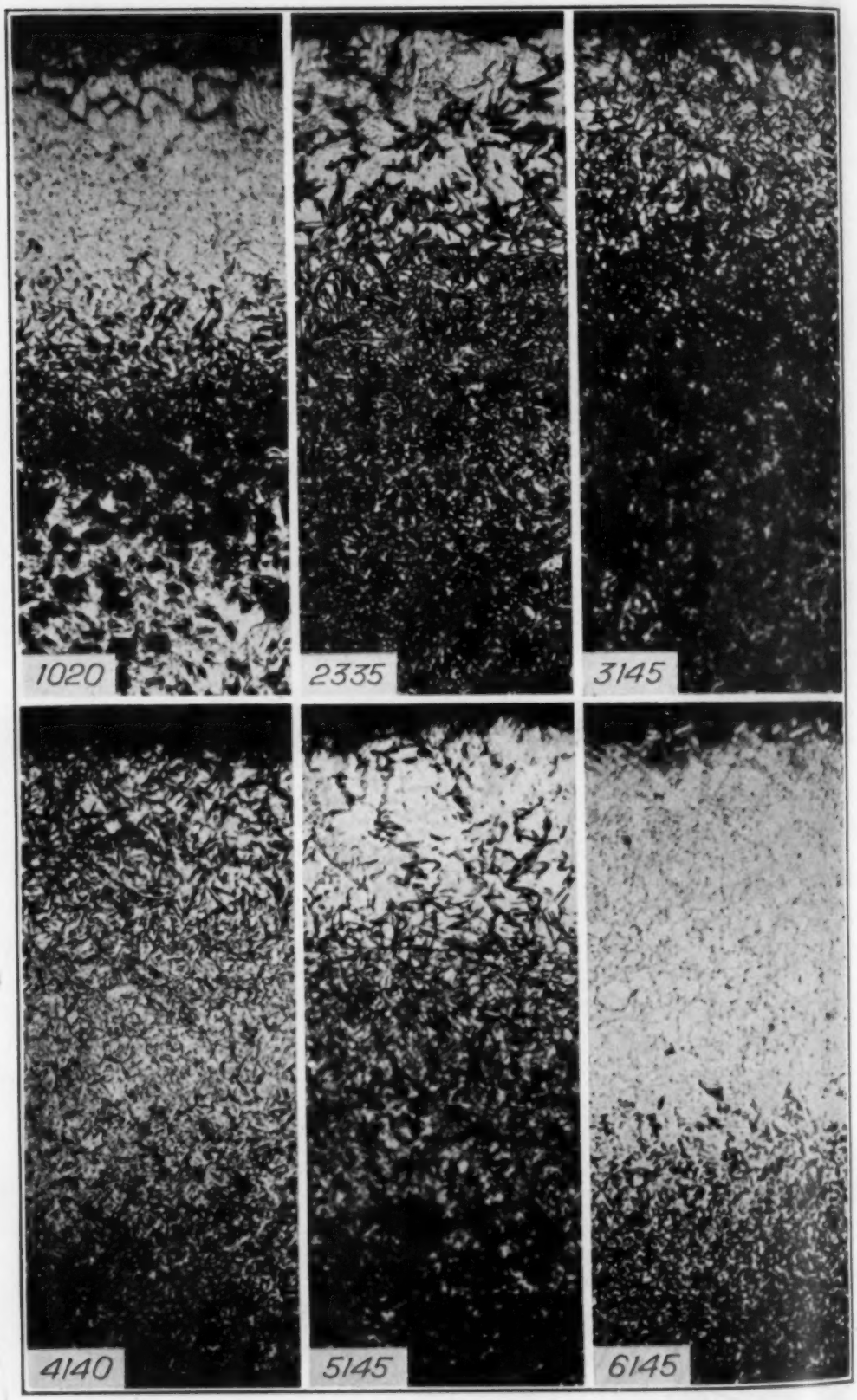

Fig. 3—Carburized S.A.E. Steels. City Gas Plus Ammonia. Temperature 1450 Degrees Fahr. Oil-Quenched.

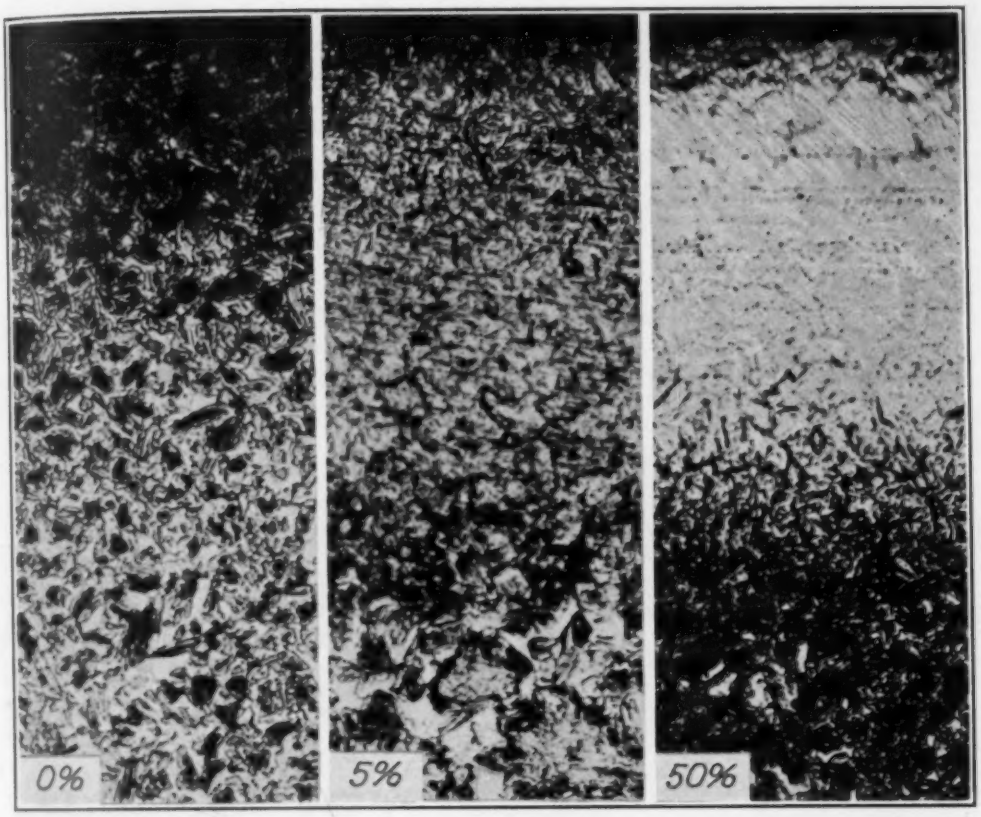

Fig. 4—Effect of Ammonia in Carburizing S.A.E. 1020 Steel. Quenched in Oil.

the same temperature in an atmosphere composed of natural gas and ammonia containing 50 per cent of each.

Third, another series of the same steels given the same treatment except that the atmosphere was Toledo city gas and ammonia—50 per cent of each.

The photomicrographs showing the results obtained are given in Figs. 1 to 3.

An inspection of these three tests reveals the important effect that ammonia has upon the carburizing reaction at comparatively low carburizing temperatures. This effect is most marked in the S.A.E. 1020 steels, but it carries through the other steels in a manner but slightly less effective. It is noticeable first of all in the case depth. It is noticeable also in the increase of hardness that is apparent from an examination of the quenched structures as revealed by the photomicrographs. This hardness is combined with wear-resistance typical of a cyanide case.

Attention is called to the fact that the effects produced by ammonia upon the case depth and microstructures are not propor-

tional to the amount of ammonia used. A small amount of ammonia has an effect upon the case depth that is about the same as that produced by a larger amount of ammonia although the larger amount of ammonia produces a different type of case. This is clearly shown by reference to Fig. 4.

Second Series

A second series of tests were carried through to determine the effects of different carburizing atmospheres in conjunction with different amounts of ammonia, upon the type of case produced, and the amount of carbon and nitrogen produced in the case as determined by the analysis of successive cuts taken throughout the case depth. The following tests were run:

1. Natural gas CG + natural gas + 25 per cent ammonia.
2. Natural gas CG + natural gas + ammonia 5 per cent.
3. Natural gas CG + 25 per cent ammonia.
4. Natural gas + DX gas + 25 per cent ammonia.
5. Natural gas + DX gas + 25 per cent ammonia—temperature 1350 degrees Fahr.
6. Same gas as 5—temperature 1550 degrees Fahr.
7. Same gas as No. 5 but 5½ hours at 1450 degrees Fahr.

A series of steels were run in each of these tests. The photomicrographs, Figs. 5 and 6, show the results obtained when using S.A.E. 1020 steel. The specimens have all been slowly cooled from a temperature of 1450 degrees Fahr., except as noted. In the caption of each photomicrograph is given the carbon and nitrogen analysis with successive cuts of 0.004 inch. To show these results graphically, they are plotted and reproduced as Figs. 7 to 13. Figs. 14 to 16 show the results on tests previously referred to.

It is to be noted that ammonia can be used in conjunction with any of the usual carburizing gas mixtures. By varying these mixtures along with ammonia it is possible to vary the carbon and nitrogen contents of the case which indicates that a proper selection of gases will enable one to raise or lower the carbon or nitrogen content of the case as may be desired. This makes it possible to determine the type of case required and to reproduce this consistently by means of a control of the gas mixtures and temperatures used. It is to be noted also that the temperature has an effect upon the case concentration and also that it is possible by this means to produce rather deep cyanided cases of definite composition.

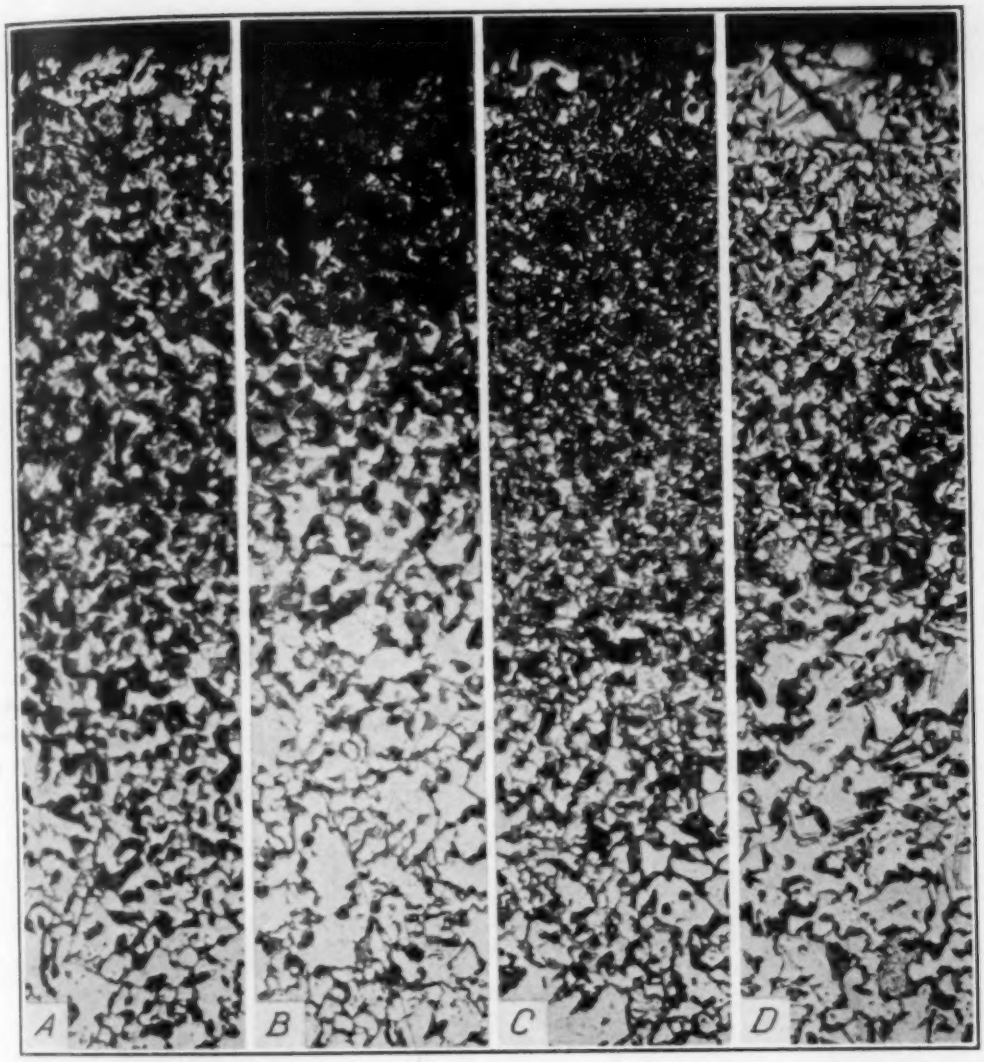

Fig. 5—Effect of Various Carburizing Gases with Ammonia in Carburizing S.A.E. 1020 Steel. 1450 Degrees Fahr. Slowly Cooled. × 300. Case Gradient 0.004 Inch Layers.

Fig. 5A—Test No. 1—25% NH_3. Nat. Gas CG + X-Nat. Gas + Ammonia. 1. 0.88 C—0.31 N_2. 2. 0.50 C—0.14 N_2. 3. 0.28 C—0.02 N_2. 4. 0.23 C—0.019 N_2.

Fig. 5B—Test No. 2—5% NH_3. Nat. Gas CG + X-Nat. Gas + Ammonia. 1. 0.87 C—0.13 N_2. 2. 0.55 C—0.08 N_2. 3. 0.33 C—0.011 N_2. 4. 0.25 C—0.011 N_2.

Fig 5C—Test No. 3—25% NH_3. Nat. Gas CG + Ammonia. 1. 0.78 C—0.33 N_2. 2. 0.52 C—0.28 N_2. 3. 0.32 C—0.09 N_2. 4. 0.25 C—0.031 N_2.

Fig. 5D—Test No. 4—25% NH_3. Nat. Gas + Dx Gas + Ammonia. 1. 1.03 C—0.67 N_2. 2. 0.64 C—0.28 N_2. 3. 0.34 C—0.051 N_2. 4. 0.24 C—0.049 N_2.

Third Series

This series was carried through for the purpose of revealing the effects of different temperatures upon a variety of S.A.E. steels. The results obtained are shown in the photomicrographs Figs. 17 to 23 inclusive. The test numbers in the captions of the photomicrographs are the same as those used in the Second Series to which reference can be made for details of the atmospheres used. Attention is

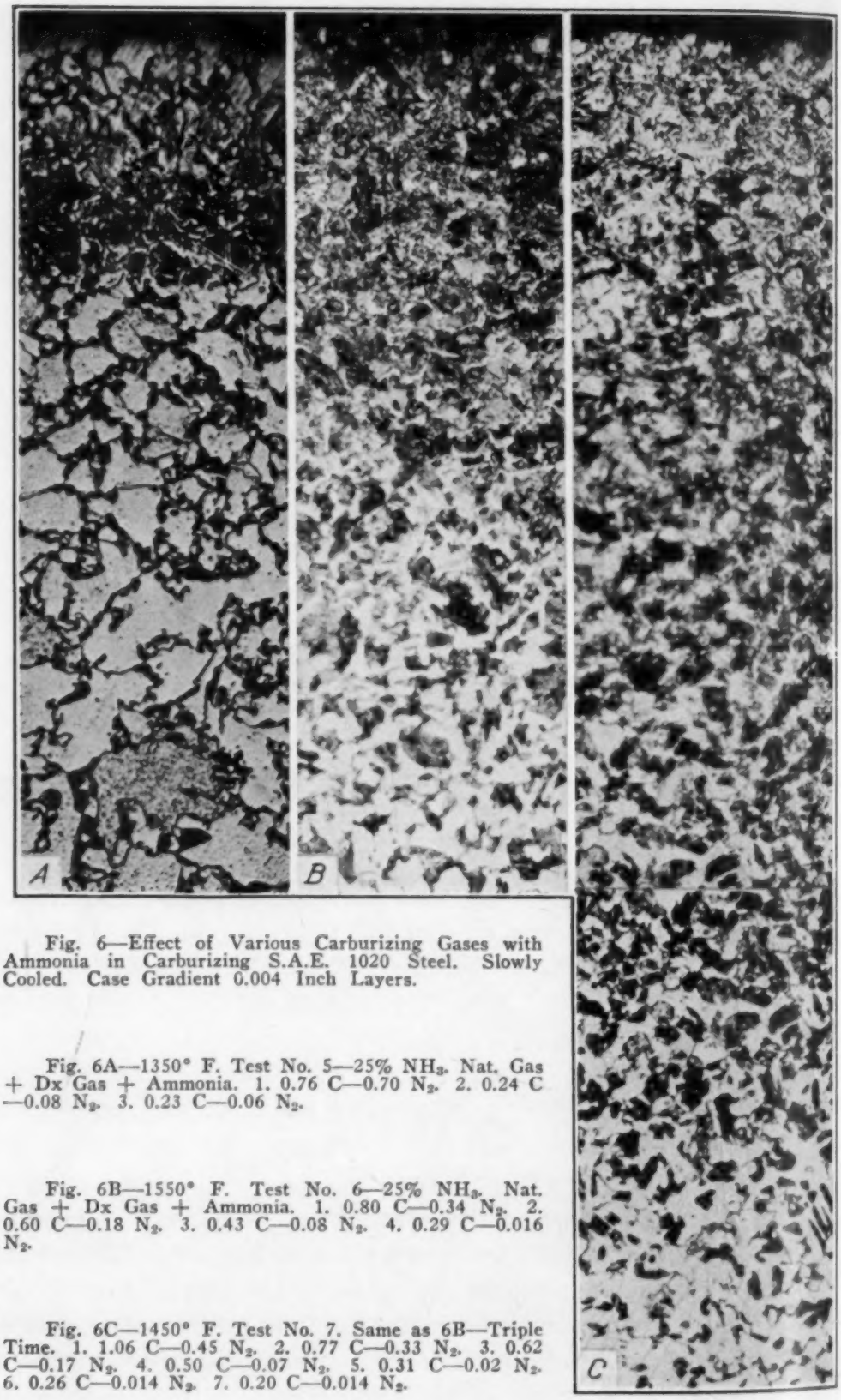

Fig. 6—Effect of Various Carburizing Gases with Ammonia in Carburizing S.A.E. 1020 Steel. Slowly Cooled. Case Gradient 0.004 Inch Layers.

Fig. 6A—1350° F. Test No. 5—25% NH_3. Nat. Gas + Dx Gas + Ammonia. 1. 0.76 C—0.70 N_2. 2. 0.24 C —0.08 N_2. 3. 0.23 C—0.06 N_2.

Fig. 6B—1550° F. Test No. 6—25% NH_3. Nat. Gas + Dx Gas + Ammonia. 1. 0.80 C—0.34 N_2. 2. 0.60 C—0.18 N_2. 3. 0.43 C—0.08 N_2. 4. 0.29 C—0.016 N_2.

Fig. 6C—1450° F. Test No. 7. Same as 6B—Triple Time. 1. 1.06 C—0.45 N_2. 2. 0.77 C—0.33 N_2. 3. 0.62 C—0.17 N_2. 4. 0.50 C—0.07 N_2. 5. 0.31 C—0.02 N_2. 6. 0.26 C—0.014 N_2. 7. 0.20 C—0.014 N_2.

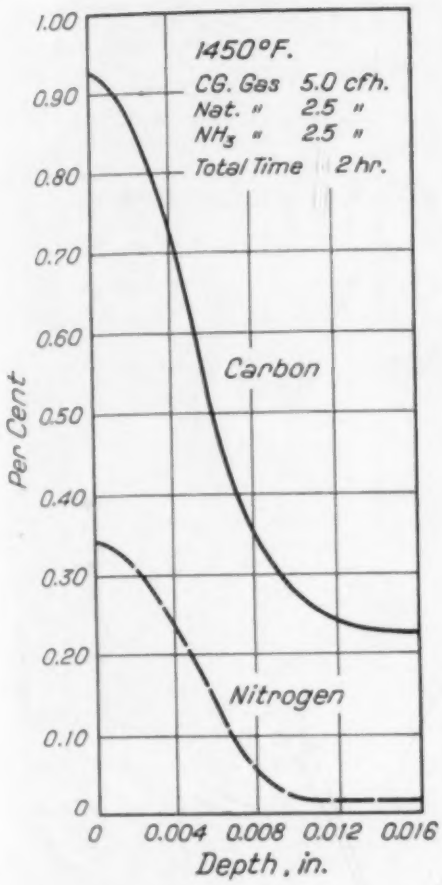

Fig. 7—Test No. 1—Carbon and Nitrogen Penetration Curve. S.A.E. 1020 Steel.

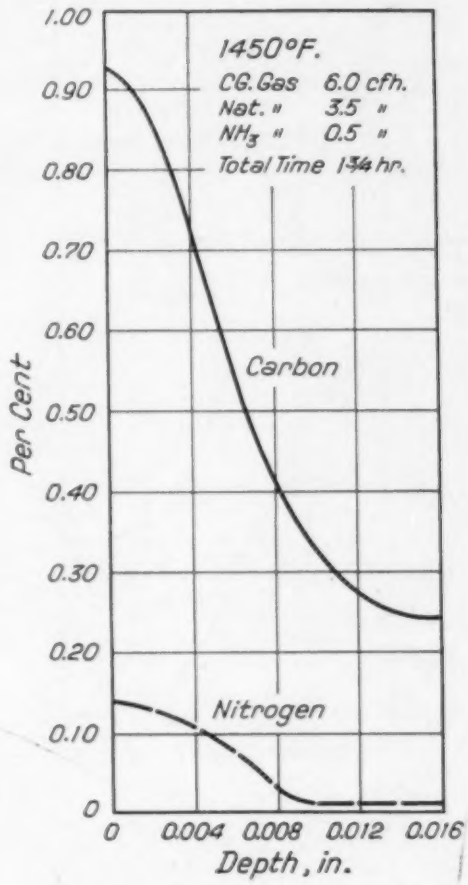

Fig. 8—Test No. 2—Carbon and Nitrogen Penetration Curve. S.A.E. 1020 Steel.

called particularly to the Rockwell hardness values given. These must be interpreted in relation to the quenching temperature and the critical point of the steel used.

The results are shown for a variety of the common steels. The effect of temperature upon the case character and depth is clearly revealed by a study of these micrographs. A critical examination of the results is very profitable. It is not necessary at this time to point out all the effects produced. It is interesting to compare the responsiveness of the different steels to this treatment and to note the effects of the different alloy constituents upon the case produced. No attempt will be made to generalize as to these results.

There was no signs of brittleness in any of the cases examined. A dynamometer test of transmission parts gave satisfactory results with no indication of spalling after running with prolonged loads.

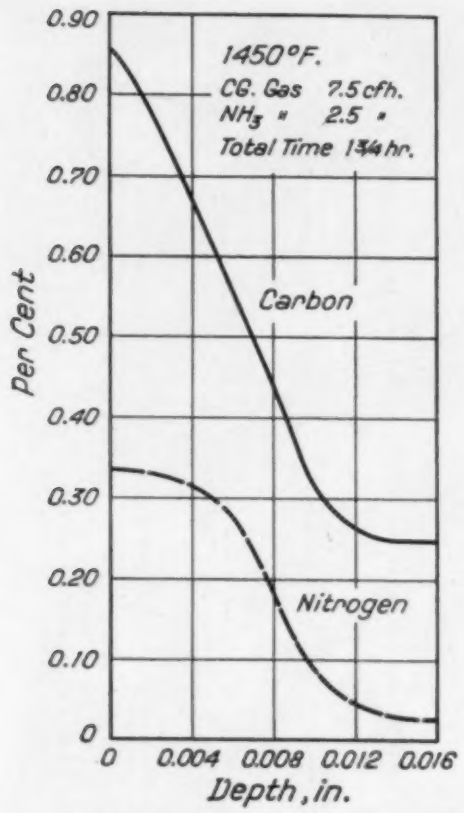

Fig. 9—Test No. 3—Carbon and Nitrogen Penetration Curve. S.A.E. 1020 Steel.

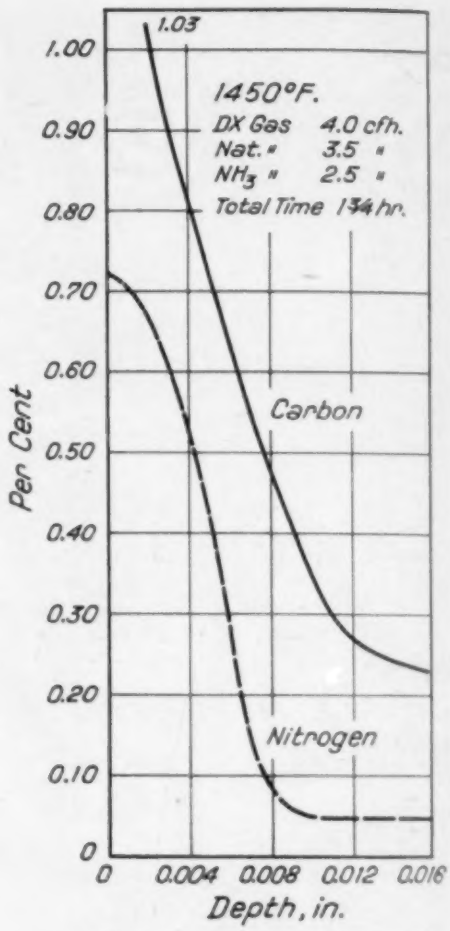

Fig. 10—Test No. 4—Carbon and Nitrogen Penetration Curve. S.A.E. 1020 Steel.

Section 4—Development of Process

In the development of this process for continuous gas cyaniding, it has been necessary to deal with a great many conflicting chemical reactions. It was soon discovered that the mere addition of ammonia to the carburizing gas was not satisfactory because of interfering reactions. This was especially true in those instances where the carburizing gas included oxygen in any form. It was found that, if the carburizing gas and ammonia were entered together in the charging end of a continuous furnace, a series of reactions took place with the formation of water vapor which then interfered with the subsequent desirable reactions and produced unsatisfactory results. This was equally true when the gases were mixed together and introduced into the muffle at a series of inlets spaced between the charging and discharging end of the furnace. This unsatisfactory reaction gave results that were very erratic, varying all the way from a deep case to

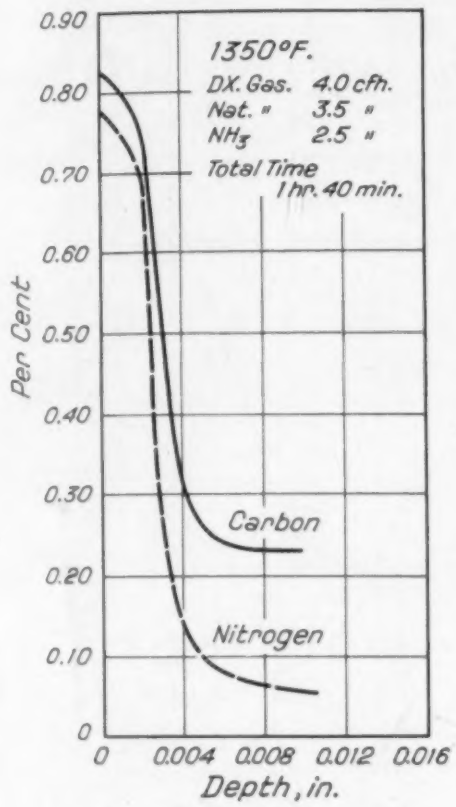

Fig. 11—Test No. 5—Carbon and Nitrogen Penetration Curve. S.A.E. 1020 Steel.

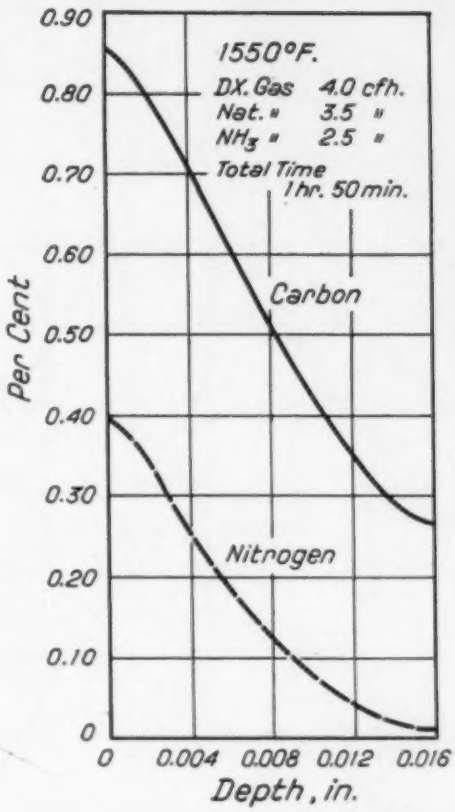

Fig. 12—Test No. 6—Carbon and Nitrogen Penetration Curve. S.A.E. 1020 Steel.

no case at all. In addition to this there were other reactions whereby carbon dioxide was formed which also produced erratic results.

The specimens obtained from these preliminary tests were all examined systematically for hardness. This was done by means of the micro-character scratch hardness test. This enabled us to determine the relative hardness of the constituents revealed by the micrographs and to identify those constituents which were of the desired hardness. This tool was of great assistance in helping to direct our efforts along the right lines.

The results obtained in these tests proved to us that it was necessary to separate the carburizing from the nitriding gases as they enter the furnace. It was found to be best to introduce the carburizing gas at the charging end of the furnace and to introduce the ammonia only at those points along the muffle where the work had come to temperature. By this means the objectionable reactions referred to above were overcome and it was possible to obtain consistent results.

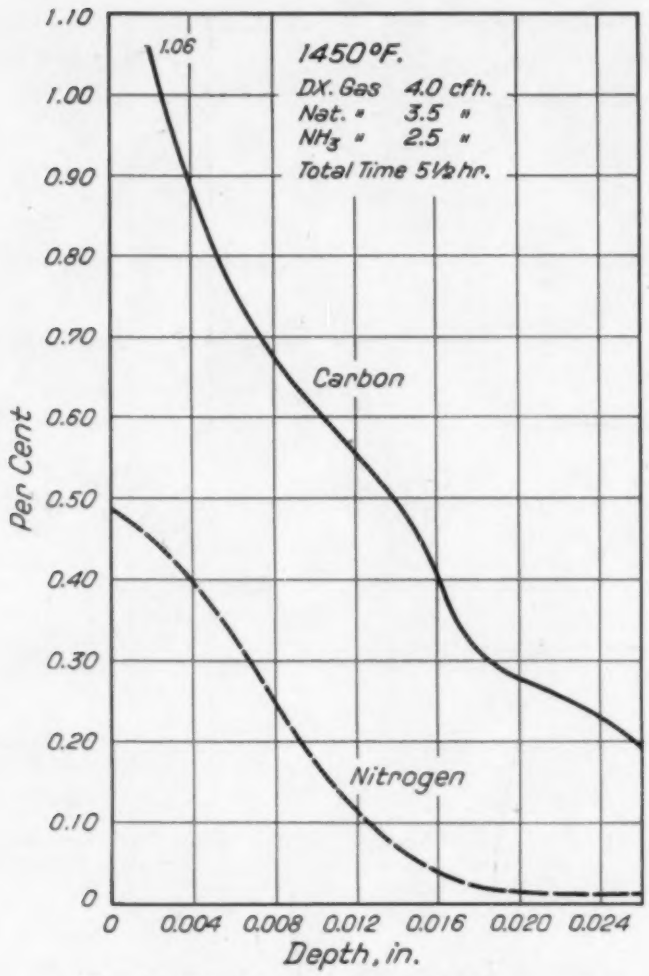

Fig. 13—Test No. 7—Carbon and Nitrogen Penetration Curve. S.A.E. 1020 Steel.

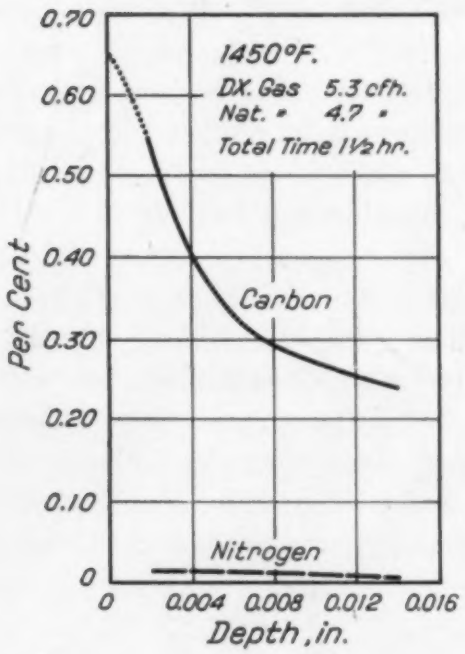

Fig. 14—Test No. 8—Carbon and Nitrogen Penetration Curve. S.A.E. 1020 Steel.

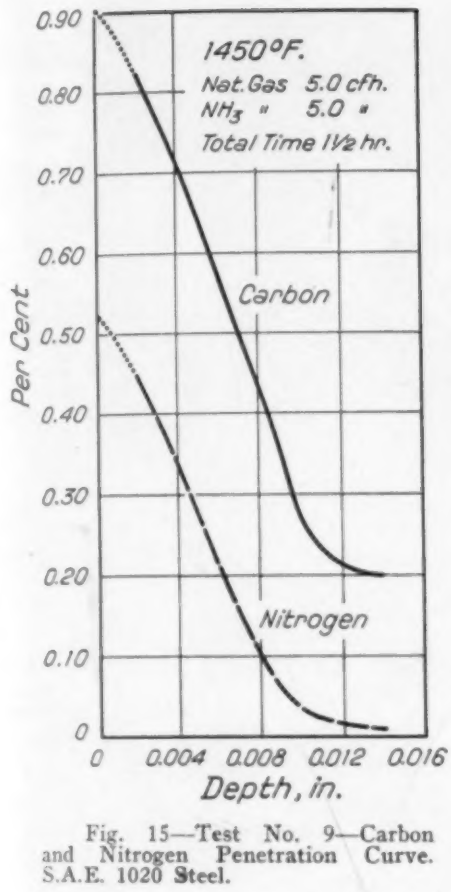

Fig. 15—Test No. 9—Carbon and Nitrogen Penetration Curve. S.A.E. 1020 Steel.

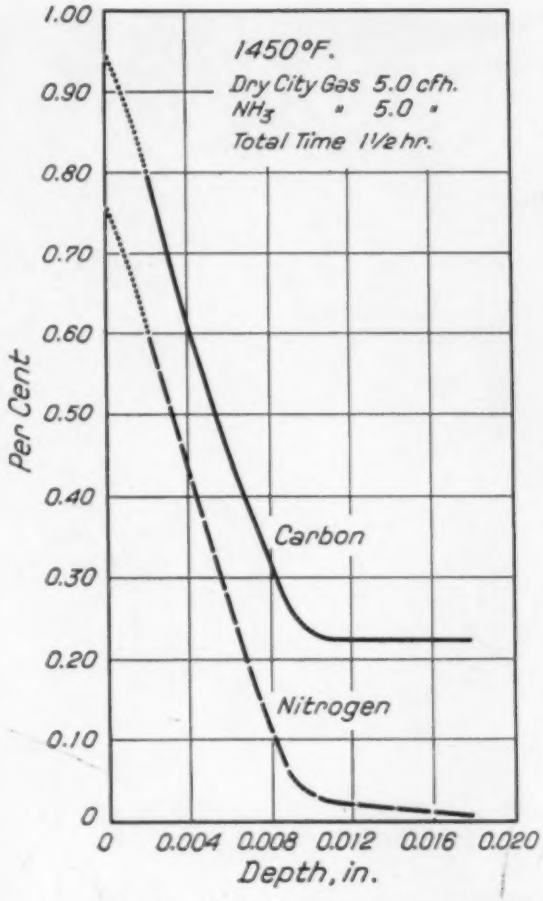

Fig. 16—Test No. 10—Carbon and Nitrogen Penetration Curve. S.A.E. 1020 Steel.

By introducing the carburizing gas at the charging end of a continuous furnace, there is brought about a condition similar to that which is found in continuous gas carburizing, when either carbon monoxide or a hydrocarbon gas is introduced at the low temperature end of the muffle, namely, the formation, on the work while coming to temperature, of a film of amorphous carbon. When this carbon reaches the full temperature zone of the furnace, it reacts with ammonia introduced at this point to form hydrocyanic acid gas at the metal surface. Being formed in this manner this gas is a most effective cementation reagent. This arrangement makes it possible also to vary the amount of ammonia gas introduced at different points along the muffle length and has an effect upon the results.

It has been known for a long time that HCN would produce a good case on steel. Tests in our laboratory with the bottled gas have proven this to be true. Because of its dangerous character and

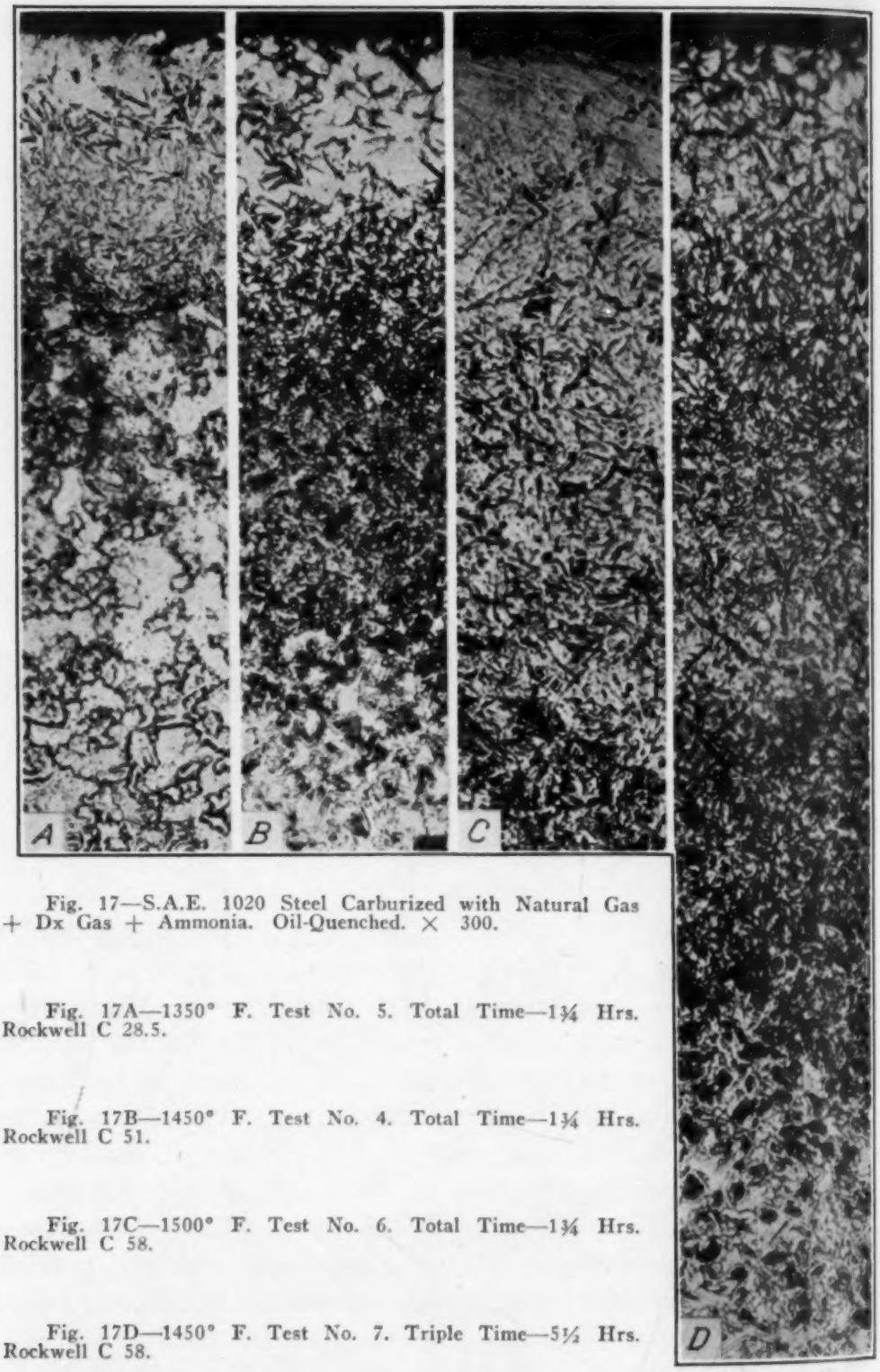

Fig. 17—S.A.E. 1020 Steel Carburized with Natural Gas + Dx Gas + Ammonia. Oil-Quenched. × 300.

Fig. 17A—1350° F. Test No. 5. Total Time—1¾ Hrs. Rockwell C 28.5.

Fig. 17B—1450° F. Test No. 4. Total Time—1¾ Hrs. Rockwell C 51.

Fig. 17C—1500° F. Test No. 6. Total Time—1¾ Hrs. Rockwell C 58.

Fig. 17D—1450° F. Test No. 7. Triple Time—5½ Hrs. Rockwell C 58.

expensiveness, it is not recommended for industrial use. When formed from its elements within the muffle itself, it is a very effec-

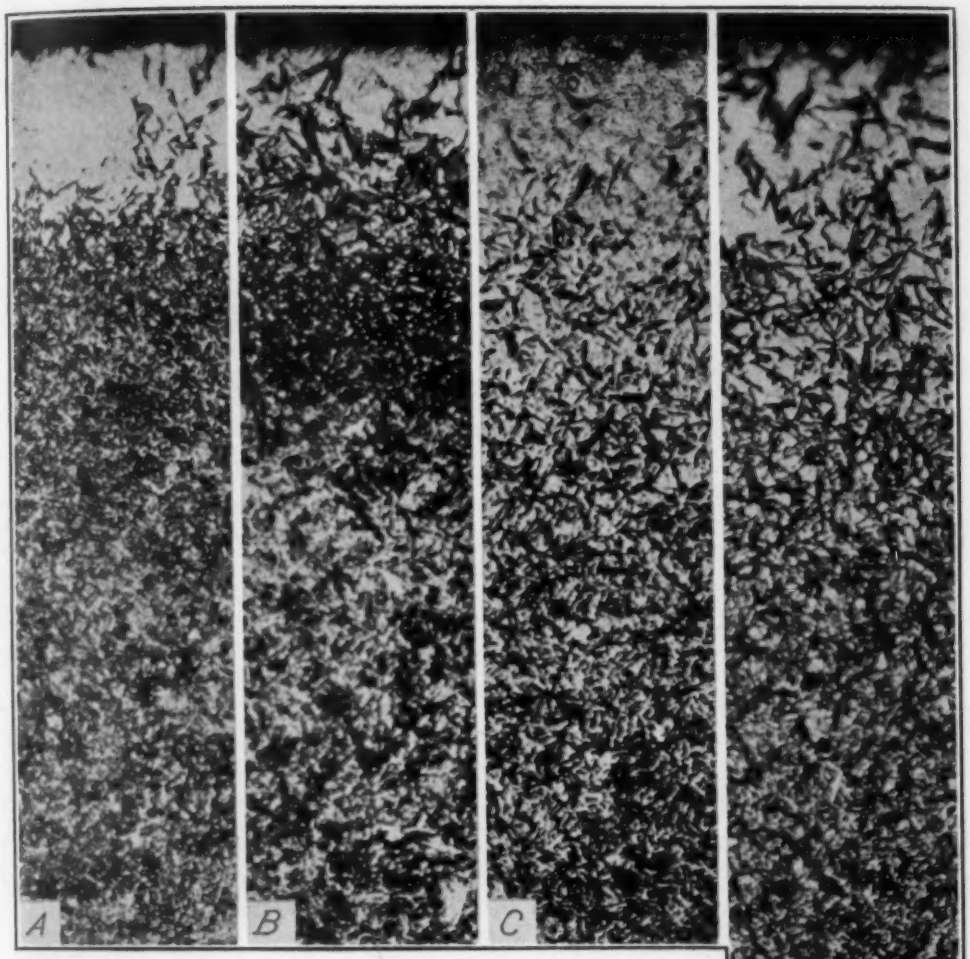

Fig. 18A and B—S.A.E. 2315 Steel Carburized with Natural Gas + Dx Gas + Ammonia. × 300.

Fig. 18A—1350° F. Test No. 5. Total Time—1¾ Hrs. Rockwell C 56.6.

Fig. 18B—1450° F. Test No. 4. Total Time—1¾ Hrs. Rockwell C 56.6.

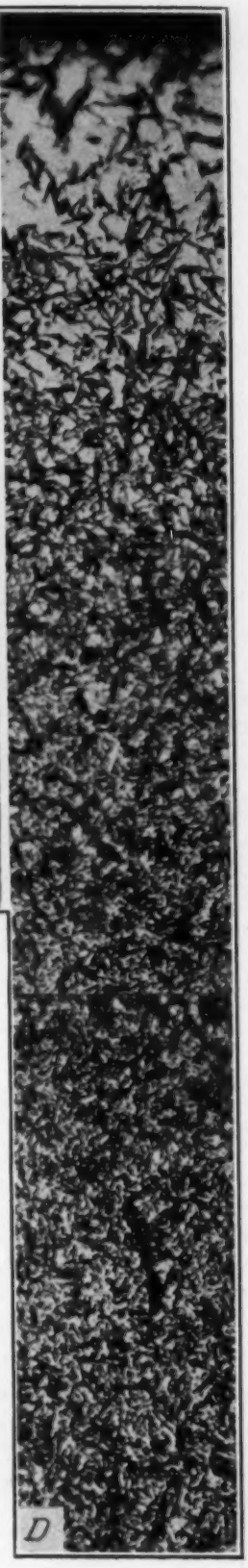

Fig. 18C and D—S.A.E. 2335 Steel Carburized with Natural Gas—Dx Gas—Ammonia. × 300.

Fig. 18C—1550° F. Test No. 6. Total Time—1¾ Hrs. Rockwell C 62.

Fig. 18D—1450° F. Test No. 7. Triple Time—5½ Hrs. Rockwell C 57.5.

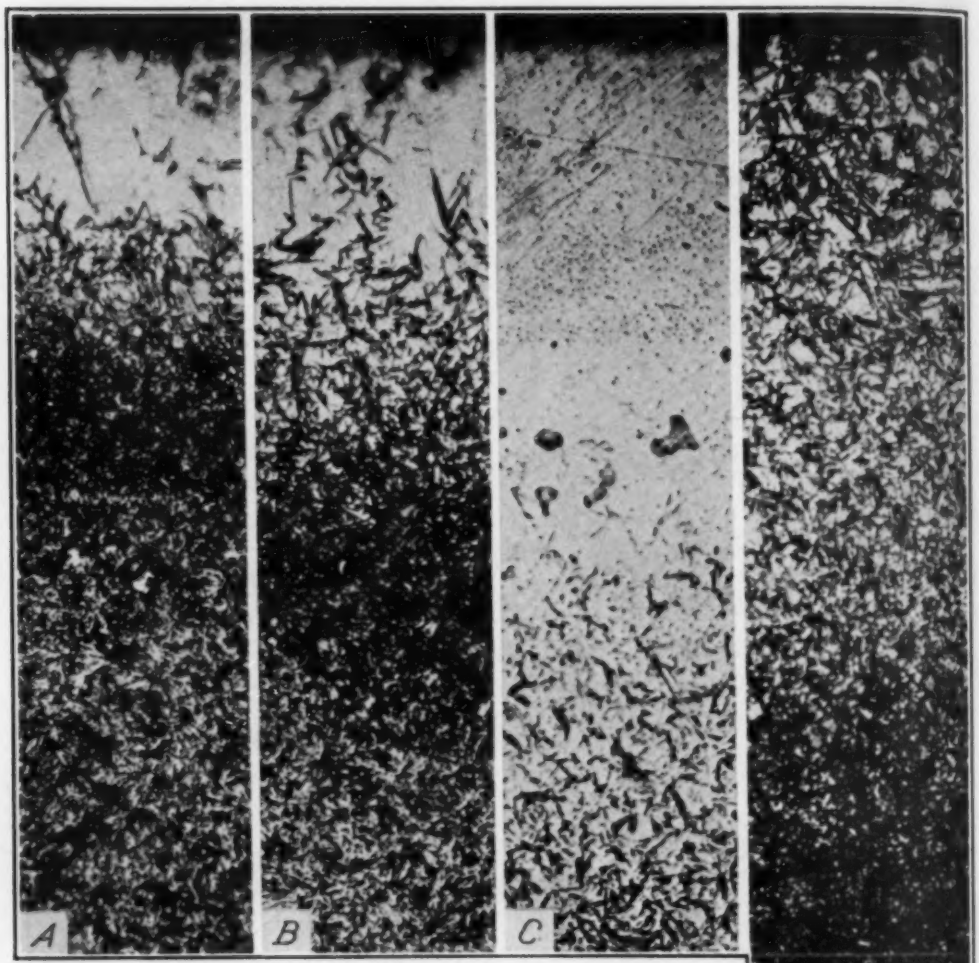

Fig. 19—S.A.E. 3140 Steel Carburized with Natural Gas + Dx Gas + Ammonia. × 300.

Fig. 19A—1350° F. Test No. 5. Total Time—1¾ Hrs. Rockwell C 58.5.

Fig. 19B—1450° F. Test No. 4. Total Time—1¾ Hrs. Rockwell C 58.

Fig. 19C—1550° F. Test No. 6. Total Time—1¾ Hrs. Rockwell C 62.5.

Fig. 19D—1450° F. Test No. 7. Triple Time—5½ Hrs. Rockwell C 59.5.

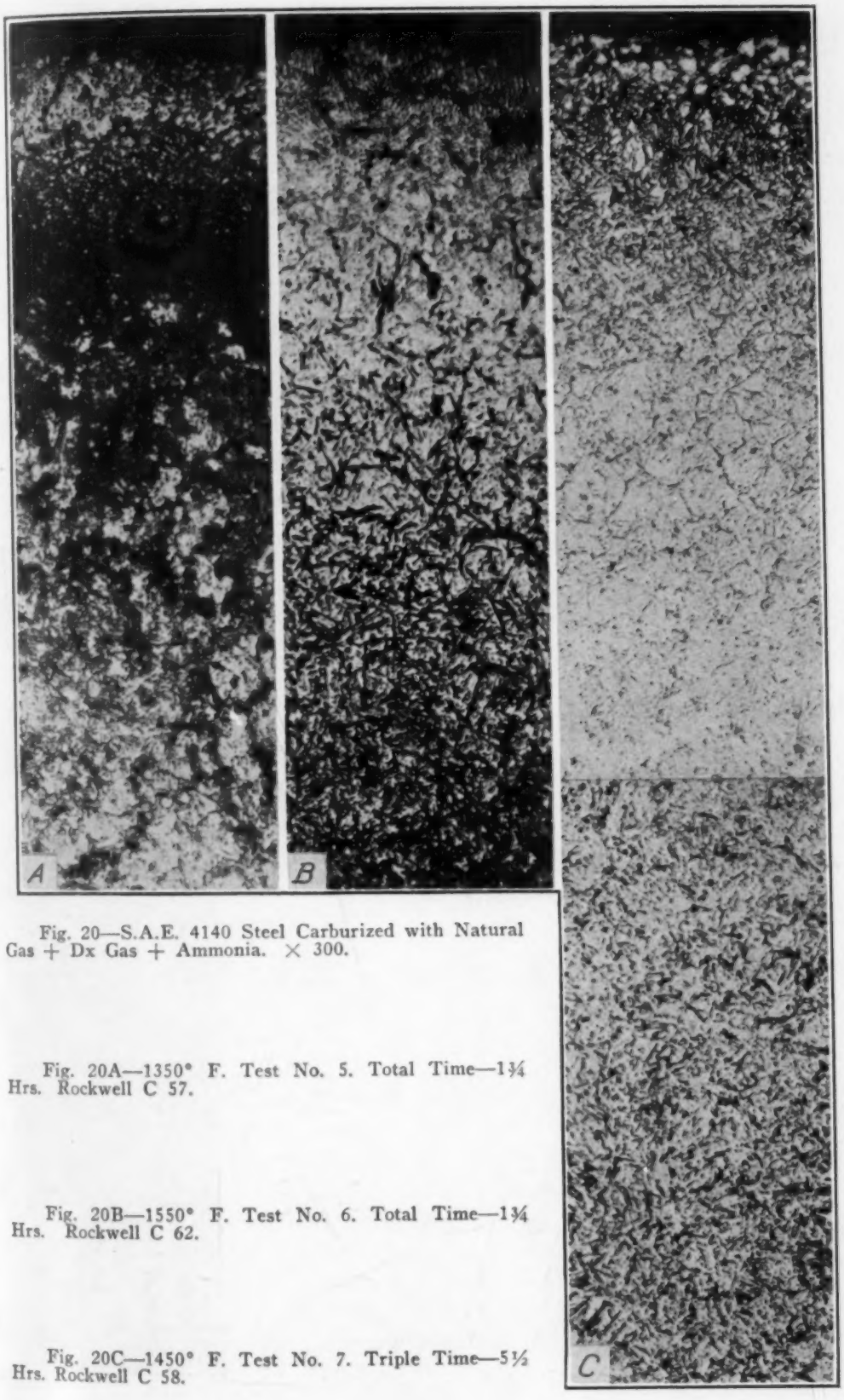

Fig. 20—S.A.E. 4140 Steel Carburized with Natural Gas + Dx Gas + Ammonia. × 300.

Fig. 20A—1350° F. Test No. 5. Total Time—1¼ Hrs. Rockwell C 57.

Fig. 20B—1550° F. Test No. 6. Total Time—1¼ Hrs. Rockwell C 62.

Fig. 20C—1450° F. Test No. 7. Triple Time—5½ Hrs. Rockwell C 58.

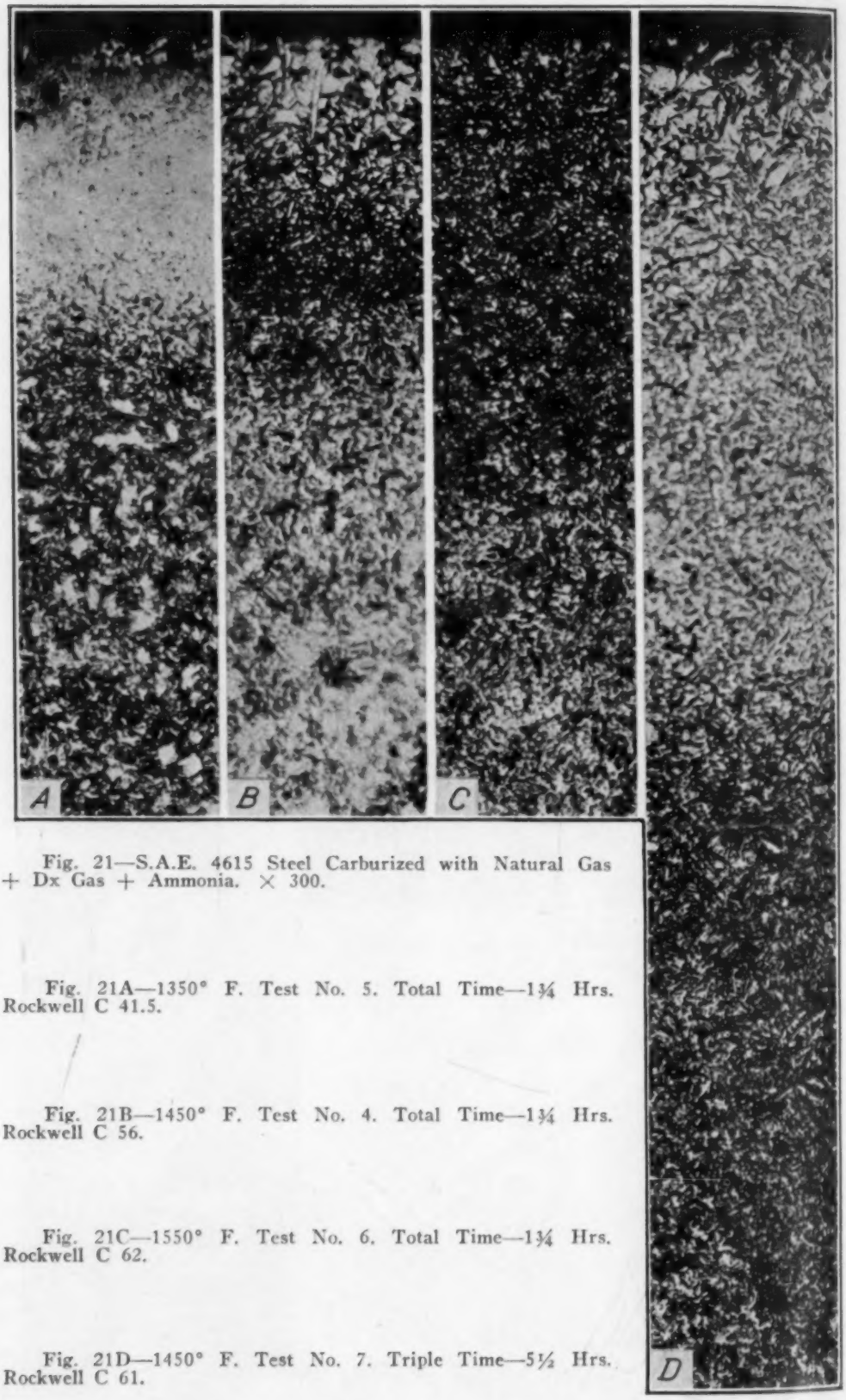

Fig. 21—S.A.E. 4615 Steel Carburized with Natural Gas + Dx Gas + Ammonia. × 300.

Fig. 21A—1350° F. Test No. 5. Total Time—1¾ Hrs. Rockwell C 41.5.

Fig. 21B—1450° F. Test No. 4. Total Time—1¾ Hrs. Rockwell C 56.

Fig. 21C—1550° F. Test No. 6. Total Time—1¾ Hrs. Rockwell C 62.

Fig. 21D—1450° F. Test No. 7. Triple Time—5½ Hrs. Rockwell C 61.

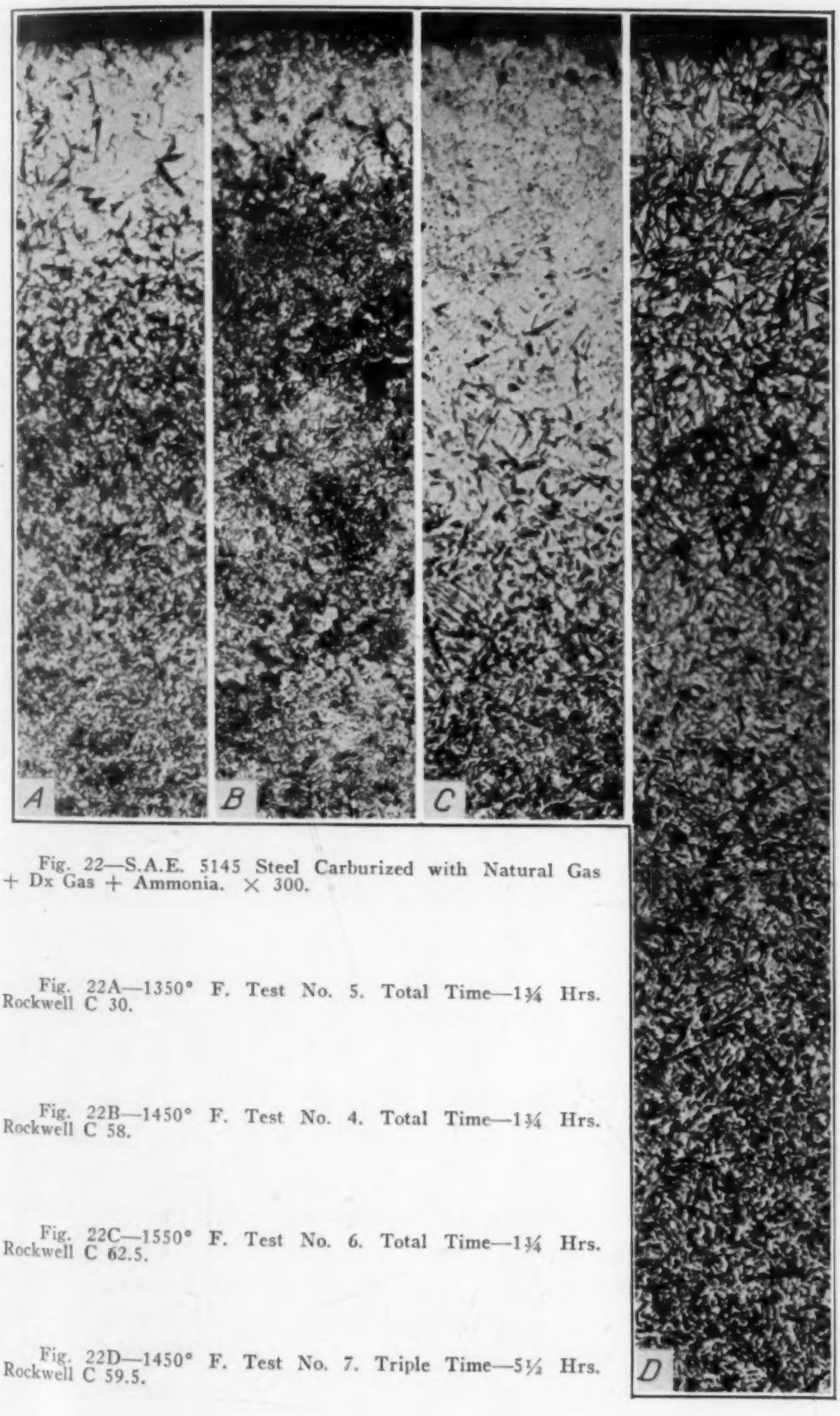

Fig. 22—S.A.E. 5145 Steel Carburized with Natural Gas + Dx Gas + Ammonia. × 300.

Fig. 22A—1350° F. Test No. 5. Total Time—1¾ Hrs. Rockwell C 30.

Fig. 22B—1450° F. Test No. 4. Total Time—1¾ Hrs. Rockwell C 58.

Fig. 22C—1550° F. Test No. 6. Total Time—1¾ Hrs. Rockwell C 62.5.

Fig. 22D—1450° F. Test No. 7. Triple Time—5½ Hrs. Rockwell C 59.5.

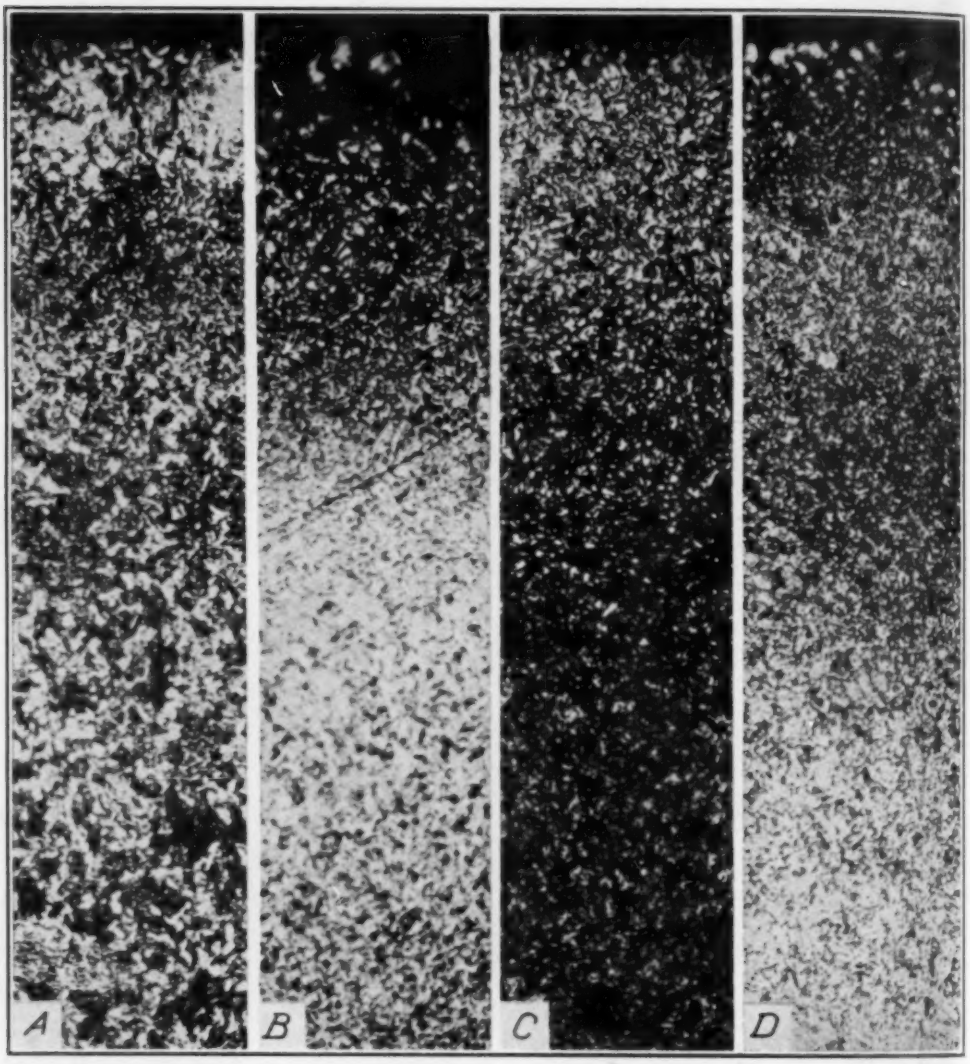

Fig. 23—S.A.E. 6145 Steel Carburized with Natural Gas + Dx Gas + Ammonia. × 300.
Fig. 23A—1350° F. Test No. 5. Total Time—1¼ Hrs. Rockwell C 28.5.
Fig. 23B—1450° F. Test No. 4. Total Time—1¼ Hrs. Rockwell C 60.
Fig. 23C—1550° F. Test No. 6. Total Time—1¼ Hrs. Rockwell C 64.
Fig. 23D—1450° F. Test No. 7. Triple Time—5½ Hrs. Rockwell C 61.5.

tive gas. This has been found to be a very important reaction especially when occurring at a metal surface. When produced in this way, there is no hazard attending its use.

The chemistry of the reactions involved in this process has been very carefully studied. The problem has been to eliminate the objectionable reactions and to facilitate the desired reactions in such a way as to have them occur at the correct time in the temperature cycle. The results that have been presented above are sufficient proof of the flexibility of this operation. Other nitrogen containing com-

pounds have been used as well as ammonia and it has been found that some of these can produce excellent results. From the standpoint of economy of operation, the use of ammonia for this purpose is by far the best.

Summary

It has been shown that the effect of ammonia upon the carburizing reaction is very marked. By means of this reaction, it is possible to carburize at temperatures very much lower than the usual carburizing operations. In fact, in one test not reported in this paper, we were able to obtain carburization at a temperature of 1150 degrees Fahr., which is in accordance with the observations made by other investigators as to the effect of nitrogen upon the critical points of the steel. Many of the specimens obtained have been found to be quite hard without a quenching operation.

It has been shown to be possible to use ammonia in conjunction with a variety of carburizing gases and that by varying these gases and the temperature of operation thereby to produce cases of different nitride and carbide compositions.

A variety of the usual S.A.E. steels have been dry cyanided at temperatures of 1350, 1450 and 1550 degrees Fahr. In each case these samples have been quenched in oil and their resulting hardnesses determined as obtained on the Rockwell scale.

In order to obtain these results, it has been necessary to introduce the gases into the muffle in a particular way and this has given rise to a new method of heat treatment which we have designated as dry cyaniding.

It is desired to mention the names of a few of the men who have been identified with the various stages of this work. Mr. John Hoffman has carried on most of the experimental work. Our thanks are due also to Mr. Orville Cullen, Mr. Harry Schwab and Mr. J. Paul Johnson. The photomicrographs were all taken by Mr. Bryce. The work has been under the general supervision of Mr. E. G. de Coriolis. This is a contribution from the laboratories of Surface Combustion Corporation.

EFFECT OF TEMPERING QUENCHED HYPEREUTECTOID STEELS ON THE PHYSICAL PROPERTIES AND MICROSTRUCTURE

By Charles R. Austin and B. S. Norris

Abstract

This paper presents a critical review on the literature dealing with reactions occurring in carbon steels below the eutectoid inversion, and with their effect on physical properties. The difference in reaction to tempering hypereutectoid carbon steels, of similar chemical composition, after quenching from 1000 degrees Cent. (1830 degrees Fahr.), is then discussed in terms of electrical resistivity, magnetization-coercive force, Rockwell hardness and microstructure. The profound difference in behavior of the steels, particularly with respect to graphitization at 650 degrees Cent. (1200 degrees Fahr.), is illustrated by the data obtained on the physical properties and by metallographic records.

Austenitic grain size in relation to thermal pretreatment and to quenching temperature is discussed.

A correlated discussion of the data, and of the factors influencing the marked difference in reaction to tempering, concludes the research.

DESPITE the importance of studies on the effects of tempering quenched hypereutectoid carbon steels the literature on this subject is very meager. In recent years much has been written on the inherent characteristics of steels as distinct from the properties associated purely with chemical composition. In particular Bain and his co-workers have clearly demonstrated the effect of grain size on the temperature and rate of reactions of the eutectoid inversion whereas Herty has given special attention to the factors controlling the separation of proeutectoid ferrite.

It is well known among metallurgists dealing with high carbon steels, that similar tempering treatments may produce widely different results in quenched eutectoid steels but there appears to be little fundamental data available which provide a clear picture of the dif-

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A paper presented before the Nineteenth Annual Convention of the Society held in Atlantic City, October 18 to 22, 1937. Of the authors, Charles R. Austin is associate professor of metallurgy and B. S. Norris is graduate assistant in metallurgical department, Pennsylvania State College, State College, Pa. Manuscript received June 24, 1937.

ferences in rates of reaction which one may be likely to encounter in the tempering of such "similar" carbon steels.

In the present paper the authors have attempted to present a study on the variation in response to tempering, of steels containing about 1 per cent carbon, after water quenching from 1000 degrees Cent. (1830 degrees Fahr.).

Some early work on tempering a hypereutectoid steel after quenching from 1000 degrees Cent. (1830 degrees Fahr.), conducted for the senior author by J. H. Kelly as a thesis in his senior undergraduate year, resulted in data which were not to be anticipated. In fact the investigation emphasized the need for a closely correlated study of the differences which might be obtained on tempering high carbon steels from the martensitic state.

The importance of the need for a better understanding of the factors which control or influence the changes which occur during tempering should need no emphasis. The immediate practical significance of the study relates to:

1. The difference in what may be termed tempering reaction rates of similar carbon steels during heat treatment and the consequent effects on the mechanical properties of the steels, and
2. The effects of these different tempering reaction rates on the high temperature properties of steels when considered for service at elevated temperatures.

Present Investigation

Preliminary tests were conducted on a hypereutectoid steel by first quenching a 14-inch specimen from 950 degrees Cent. (1740 degrees Fahr.) and then tempering for 48 hours in a furnace having a temperature gradient ranging from 450 to 850 degrees Cent. (840 to 1560 degrees Fahr.). The results obtained from a study of the hardness and microstructure of the bar revealed peculiar characteristics which appeared to merit more extensive study.

Accordingly several high carbon steels were procured of fairly similar chemical composition but prepared by different methods of manufacture and having a variation in austenitic grain size. These steels were quenched to produce martensite, and the effects of tempering for progressively increasing times at 550, 650 and 710 degrees Cent. (1020, 1200 and 1310 degrees Fahr.) were studied.

The changes during tempering were followed by means of electrical resistivity, coercive force and Rockwell hardness measurements and by metallographic studies.

Review of Literature

No comprehensive review of relevant literature is available on the subject matter of the paper, and it is proposed to devote considerable attention to previous investigations.

Ishen (1)[1] subjected a one per cent carbon steel to heat treatments which were designed to produce the carbides in various shapes and sizes. He showed that for the same tensile strength, laminated carbides gave less ductility than spheroidal carbides, and that the carbides assumed an equiaxed condition more rapidly during tempering when the steel had been previously oil-quenched from above the lower critical than when slowly cooled from the same temperature. Thus finely dispersed carbides became more rapidly equiaxed than coarse lamellar carbides.

Other investigators have observed that the condition of the steel prior to heating at temperatures below the lower critical point has a profound influence upon the rate at which the carbides spheroidize. Bailey and Roberts (2) found that the cast structure of a 0.3 per cent carbon steel required ten times as much time to produce a given spheroidized state when tempered at 675 degrees Cent. (1250 degrees Fahr.) than when normalized. Cold working reduced the time to one fiftieth of that required for the normalized condition.

Duma (3) reports that sorbitizing, lead quenching, liquid hardening and cold work treatments increase the rate of carbide spheroidization. Steels of eutectoid composition are the most sluggish and unresponsive to spheroidization. This was also observed by Bailey and Roberts (2). Alloys seem to promote rather than hinder spheroidization by virtue of their refining or sorbitizing influence upon the structure. The author, however, failed to mention the alloying elements which he had observed to behave in this manner.

Whiteley (4) found that he could produce globular pearlite, during cooling from above the lower critical temperature, without producing lamellar pearlite. This was accomplished by heating a 0.2 per cent carbon steel above the Ac_1, holding for a time insuffi-

[1]The figures appearing in parentheses refer to the bibliography appended to this paper.

cient for complete solution of the carbides in the gamma iron, cooling to and holding at a temperature located between Ae_1 (equilibrium at infinitely slow cooling) and Ar_1 for a definite time, and then quenching in water. He found that no lamellar pearlite was formed during the one hour that the specimen was held at this latter temperature. However, the undissolved carbides grew at the expense of the carbon in the gamma iron. Lamellar pearlite was formed only when the temperature was lowered below the Ar_1 critical point.

In the investigation by Kelly (5), conducted in the senior author's laboratory, it was found that when a hardened bar of 1 per cent carbon steel was tempered in a furnace having a linear temperature gradient the hardness did not decrease continuously as the temperature approached the lower critical. The results of this work will be recorded briefly in a later section of the paper.

It has been known for a number of years that steels of apparently similar chemical composition may act very differently on heat treatment. Examples which may be cited include the work of McQuaid and Ehn and others on abnormality resulting from carburizing and the development of soft spots on heat treatment, and the more recent studies on depth of hardening or hardness penetration on quenching.

Much work has been done in order to determine the factors which cause the steels to react dissimilarly, but such efforts have been directed principally to mechanism and reaction rates found in the inversion of the eutectoid gamma solid solution within its equilibrium or depressed temperature range, or at temperatures above the Ar_1 where a proeutectoid constituent separates. Bain and his co-workers (6 and 7), Shepherd (8), Herty and his co-workers (9 and 10), McQuaid (11), Rapatz and Wrazej (12) and others have done work along this line.

From their work one may conclude that the method used in making the steel may have a profound effect upon its properties. This is particularly true when considering the methods of deoxidation. High impact, shallow hardening and fine-grained steels result when metallic aluminum, within certain limits, is used for deoxidation. When ferrosilicon is used lower impact, deeper hardening and larger grains usually result. The size of the austenite grain prior to cooling appears to exert a great influence upon subsequent properties.

In his classical researches Bain postulated that submicroscopic

particles of aluminum oxide were the principal cause of the difference in behavior of the steels since they seemed to result in the production of small austenitic grains. McQuaid (11) offered the suggestion that metallic aluminum was playing a large part. However, Herty recently submitted evidence to show that metallic aluminum has very little effect upon the size of the austenitic grains, and that oxygen must be present if fine grains are to be produced. This has also been demonstrated by Brophy and Parker (13). Although the size of the austenitic grains, prior to cooling, control the properties to a large extent, instances arise where steels having coarse grains are shallow hardening. Shepherd's development of the P-F test—hardness penetration along with grain size from fracture—takes account of these discrepancies in the reaction of steels to hardening.

Smith (14) has shown that various physical properties are influenced by different factors.

Electrical resistance is primarily affected by the presence of chemical elements dissolved in the matrix of the alloy. Cleaves and Thompson (15) report that the resistivity for the purest iron now obtainable is approximately 9.8 microhms centimeter. Norbury (16) has listed the increase in resistivity of iron when one atomic per cent of various chemical elements is dissolved. His values, changed to one per cent by weight, are given by Sisco (17) along with those of Abegg.

Dissolved carbon exerts the most influence. It is approximately three times as effective as silicon, aluminum, phosphorus or nitrogen, and seven times as effective as manganese, chromium, vanadium, molybdenum, and copper for equal weights per cent. Nickel, tungsten, and cobalt are only one-twentieth as effective as carbon.

Yensen (17) has distinguished between the effect produced by the carbon dissolved in the iron and that which is out of solution. The former increases the resistance twenty times as much as the latter for a given weight per cent. According to Hume-Rothery (18) the increase in resistance is greater as the solute and solvent elements lie farther apart in the periodic table. Although the solution of a single chemical element in a solvent metal produces an increase in the electrical resistance of the solvent, it does not follow that the increase produced by the addition of two or more elements is additive.

The size of the ferrite grains of pure iron has an effect upon the resistivity. Thompson (19) found that he could change the resistance by approximately one microhm by increasing the number of grains.

In steels, this effect may be overshadowed by the influence exerted by the added elements.

Heat treatment has a profound effect upon electrical resistance. Campbell (20) showed that increase in tempering temperature progressively decreased the resistance of quenched steels, and concluded that the decrease was due to the precipitation of carbon from alpha iron. Bardenhauser and Schmitt (17) observed that a structure composed of lamellar pearlite had a higher resistance than one consisting of spheroidal carbides.

The temperature coefficient of resistance is much less for steels than it is for pure iron. Gumlich (17) found that it was larger when the steels were slowly cooled from the critical range than when rapidly cooled. It decreased as the carbon content increased up to 1 per cent. The coefficient falls from 0.0059 for pure iron, to 0.0043 for a slowly cooled 1 per cent carbon steel, and to 0.0016 when the steel is quenched from 850 degrees Cent. (1560 degrees Fahr.).

In the past, studies have been reported in which the magnetic properties of the materials were used as a means of following structural changes during heat treatment. In most of the cases the investigators were primarily interested in determining the conditions under which certain phases would transform. Little attention was paid to changes in size and shape of the disperse phases. While working with magnetic iron oxide Gottschalk (23) found that there was a linear relationship between coercive force and the surface of the magnetic mineral particles. He postulated that a similar relationship might hold for a dispersed phase present in a metallic system.

Carbon, hydrogen and oxygen, when dissolved in iron, are known to increase the induction coercive force while silicon has little effect. The coercive force of pure iron-carbon alloys is increased from 0.17 to 1.0 oersteds, by raising carbon content from 0.0015 to 0.174 per cent. The largest changes per 0.001 per cent carbon are produced when the carbon is dissolved in the iron. In contrast to carbon, silicon up to 5 per cent induces a change in coercive force of only 0.20 oersteds. Yensen (15) has offered an explanation for this difference. He suggests that substitutional elements cause little strain in the crystal lattice and therefore little change in magnetic properties, whereas interstitial elements lead to marked lattice strain and therefore have a pronounced effect on such properties.

The size of the ferrite grain also has an appreciable effect upon the coercive force of pure iron. Thompson (19) has been able to

increase the coercive force from 2.0 to 5.0 gausses by increasing the number of ferrite grains from 276 to 690 per centimeter length.

Yensen (17) has investigated the effect of large amounts of carbon upon the coercive force of pure iron-carbon alloys. Increasing the carbon from 0.008 to 0.981 per cent caused a corresponding increase in coercive force from 0.58 to 5.60 oersteds. This difference represents the minimum effect of carbon obtained by slowly cooling the alloys. Quenching materially affects these results and Jovall (17) found that as the quenching temperature was increased above the lower critical the coercive force of a chromium ball-bearing steel was increased. The maximum value was obtained at 800 degrees Cent. (1470 degrees Fahr.). Gumlich (17) also noted a decrease in coercive force on quenching hypereutectoid steels above 800 degrees Cent. (1470 degrees Fahr.).

Koster (17) observed that a higher value was obtained for coercive force when a 0.06 per cent carbon steel was slowly cooled from the tempering temperature than when rapidly cooled. This was true until the temperature exceeded 500 degrees Cent. (930 degrees Fahr.). At higher temperatures, the rapidly cooled steel had the greater coercive force.

Mathews (21) believed that the coercive force increases as the amount of austenite, retained on quenching, increases.

Gottschalk (22 and 23) and other workers at the U. S. Bureau of Mines have recently used the property "magnetization coercive force" for studying magnetic materials. They found that a linear relationship obtained between coercive force and the surface area of the magnetic mineral particles. Davis and Hartenheim (24) have described a convenient form of apparatus for determining magnetization coercive force.

Graphitization has long been known to occur in high carbon steels during tempering, but the amount of information present in the literature is small.

Green (25) observed that a black area appeared in some hypereutectoid steel bars when they were annealed just under the lower critical. One of the crucible made steels had a chemical composition of 1.16 per cent carbon, 0.30 per cent manganese, 0.25 per cent silicon, 0.024 per cent phosphorus and 0.02 per cent sulphur. A black area developed on tempering between 704 and 724 degrees Cent. (1300 and 1335 degrees Fahr.). A light area, which was present at the center of the bar, was surrounded by the black area which in turn was sur-

rounded by a circumferential light zone. Carbides were found in the light areas but were absent in the dark one where complete graphitization had been effected by the heat treatment.

Emmons (26) has observed the presence of graphite in tool steels when they were subjected to temperatures below the Ar_1 point. He noticed that the graphite had formed from the cementite *in situ*. Portevin and Chevenard (27) have reported graphitization in a 1.6 per cent carbon, 0.28 per cent silicon and 0.16 per cent manganese steel when it was heated below the Ar_1 point.

Kinzel and Moore (28) found graphite even in a 0.15 per cent carbon, 0.20 per cent silicon, 0.49 per cent manganese, 0.023 per cent sulphur and 0.015 per cent phosphorus steel after it had been used in an oil still for three years at a temperature well below the eutectoid.

Mathews (29) has stated that he found graphitization to be a maximum in a bar subject to this phenomenon, upon heating between about 500 degrees Cent. and the lower critical.

Schwartz (31) has summarized the effect of various chemical elements upon the rate of graphitization in malleable irons. He states that small amounts of aluminum, silicon, nickel, titanium, cobalt and copper accelerate graphitization, the effects of cobalt and copper being small, while chromium, manganese, molybdenum and tin retard it.

The presence of graphite in steel is usually considered to be an undesirable condition, but recently (32) a steel was described which had partial graphitization purposely induced. It was claimed that such steels give wearing properties that are similar to gray iron and at the same time a strength comparable to steel.

Measurement of Physical Properties

As previously indicated the changes occurring during the tempering of the quenched steels were followed by means of electrical resistivity and coercive force measurements, and by hardness determinations using a Rockwell machine.

Specific Electrical Resistance. Specimens 5½ inches long and 0.25 ± 0.001 inches in diameter were used for the electrical resistance measurements. The resistance was obtained by the voltmeter-ammeter method. A specimen was placed across fixed knife edges which were four inches apart. Approximately 2 amperes direct current were passed through the specimen, the current being read accurately to 0.01 amperes. While the current was passing, the potential drop

across the knife edges was measured by use of a L and N Type K potentiometer. Current and voltage readings were taken on three positions of the bar with respect to the knife edges. The mean values were used to calculate the specific resistance. Slight changes produced by fluctuations in room temperature appeared to fall within the experimental error.

Magnetization Coercive Force. When direct current is passed through a coil of wire, a magnetic field (H) is produced within the coil, and when the magnetic substance, such as iron, is placed in the coil, the strength of the field is increased. This new value for the field strength is called flux density (B).

By plotting values of "B" against those of "H" which have been obtained from a complete magnetic cycle the familiar hysteresis loop is obtained. Fig. 1 shows two hysteresis loops. The one formed by

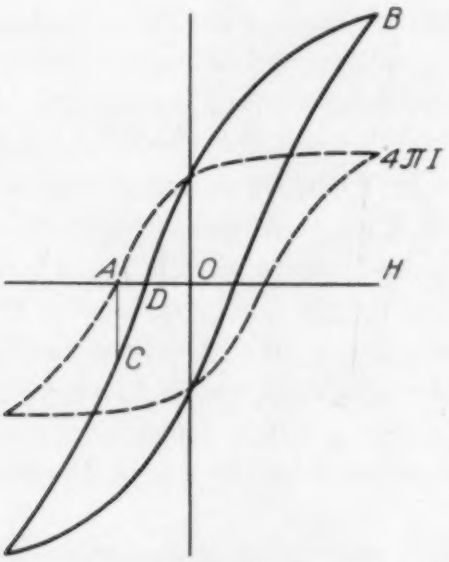


Fig. 1—Induction and Magnetization Hysteresis Loops for a Ferro-Magnetic Substance.

the solid line is called the induction hysteresis loop and is obtained by plotting flux density (B) against field strength (H). Length OD represents the value of the inductive coercive force.

The loop outlined by the dotted line is called the magnetization hysteresis loop. In this case 4π times the intensity of magnetization (I) is plotted against the field strength (H), and the distance OA represents the magnetization coercive force. The intensity of mag-

netization (I) is related to flux density (B) and field strength (H) by the formula

$$I = \frac{B - H}{4\pi}$$

According to the most recent definitions of magnetic terminology (33) the field strength is stated in oersteds while flux density and intensity of magnetization ($4\pi I$) are expressed in gausses.

A schematic diagram of the apparatus (after Davis and Hartenheim) which was used in the present investigation for measuring magnetization coercive force is shown in Fig. 2. B is the primary coil wound on a glass tube A, and connected to a source of primary current through the terminals F_1 and F_2. The secondary coil (C) is connected to the terminals E_1 and E_2 on the support D_1. These terminals are connected to a suitable ballistic galvanometer.

In order to examine the magnetic properties of a sample of steel the specimen is first placed in a solenoid and magnetized to approxi-

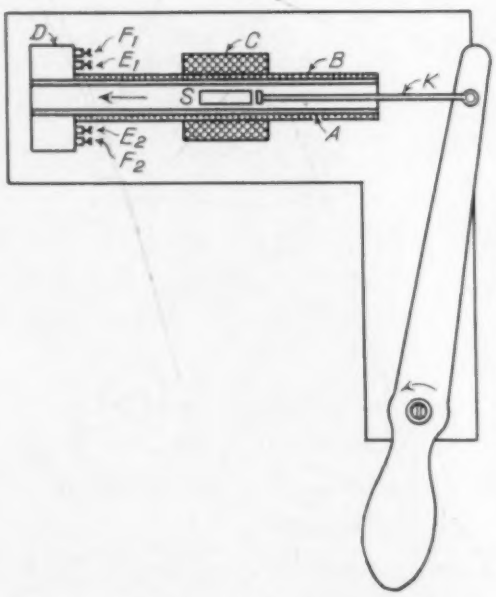


Fig. 2—Apparatus Used to Measure Magnetization Coercive Force.

mate saturation. It was experimentally found that a field strength of 450 oersteds was sufficient for the purpose with the high carbon steels investigated.

The magnetized specimen (S) is placed in the magnetic field produced by the primary coil. When the specimen is rapidly ejected

from the coil by the ram (K) an electromotive force is induced in the secondary coil whenever the magnetic field of the specimen is different from that of the primary coil. The throw of the ballistic galvanometer is directly proportional to the induced voltage. The field strength of the primary coil which exactly neutralizes the field produced by the specimen, corresponds to the magnetization coercive force. Instead of adjusting the strength of the primary current until no deflection is produced in the galvanometer, the field strengths which produced small deflections on each side of the zero point are plotted against corresponding deflections. The place where the plot crosses the zero deflection line (A Fig. 1) will correspond to the magnetic field (OA) which exactly balances that of the specimen.

The strength of this magnetic field in oersteds is calculated from the formula:

$$H = \frac{4\pi\ NC}{10 \times L}$$

where

N = turns of wire in primary coil (297)
C = amperes passing through primary coil
L = length of primary coil (27 cms.)

Thus $H = 13.82C$ oersteds.

The current (C) was of the order of 1 to 3 amperes and was measured to an accuracy of $\pm$ 0.01 amperes.

The resistivity specimens were used for the magnetic tests, and coercive force values checked to within 0.5 oersteds. The changes induced by small variations in room temperature fall within experimental error.

Hardness and Metallography. Specimens ½ inch long and ⅜ inch diameter were cut in half transversely after heat treatment. One half was used for hardness determinations while the other half was used for metallographic study. In preparation for the hardness test the cut face was ground down to 0 emery paper. The back of the specimen was also prepared to insure a flat, smooth surface. Prior to hardness determinations the Rockwell hardness tester was constantly checked with a standard block, and any necessary corrections made of the observed data.

On the other half of the specimen a metallographic study was made and records were prepared for inclusion in the section of the paper devoted to this phase of the investigation.

Data on Steels Tested

The chemical analysis of the steel, designated X, on which the preliminary tests in a gradient furnace were conducted is given in Table I. In order to study more completely the possible dissimilar reaction on tempering of several carbon steels of approximately the same chemical analysis, six other steels were obtained by the kind cooperation of three steel companies. These are designated by the letters A to F and the chemical analyses are recorded in Table I.

Table I
Chemical Composition

Type of Steel	Steel	Per Cent C	Mn	Si	P	S	Cr	Ni	Cu	Al	Al_2O_3
O.H.	A	1.07	0.37	0.26	0.017	0.034	0.06	0.06	0.13	0.039	0.012
E.F.	B	1.06	0.27	0.48	0.023	0.022	0.08	0.03	....	0.022	0.010
E.F.	C	1.12	0.25	0.30	0.019	0.013	0.11 (0.23)	0.07	....	0.020	0.018
O.H.	D	0.99	0.43	0.18	0.018	0.036	0.08	0.09	....	0.020	0.026
O.H.	E	1.04	0.46	0.215	0.017	0.037	0.04	0.08	0.10	0.020	0.028
E.F.	F	0.97	0.32	0.28	0.012	0.014	0.04	0.14	....	0.041	0.036
O.H.	X	1.05	0.34	0.16	0.02	0.031	....	....	....	.....	.....

Steels A, D, E and X are open-hearth steels, C and F electric furnace steels, and B an induction furnace steel. Steels B and C had been given a carbide spheroidizing treatment after rolling, Steel A had been softened at 680 degrees Cent. (1255 degrees Fahr.), with no spheroidizing, and Steels D, E, F, and X were received in the hot-rolled condition.

The general chemical analyses were supplied by the companies submitting the steel samples but the authors are indebted to the Republic Steel Corporation for all the analyses for aluminum and alumina, and to the Carpenter Steel Company for the chromium and nickel content of some of the steels. The grain size of the steels was also submitted. Steels A, D, E, and F had been given the A. S. T. M. test while the Shepherd test (quenched from 1550 degrees Cent.) was used for Steels B and C. The values were as follows:

Table II

Steel	Manufacture	Grain Size Number	Method of Determination
A	Open-Hearth	5–8, 15% 3–4	A.S.T.M.
B	Induction Furnace	6	Shepherd (1550 Degrees Cent.)
C	Electric Furnace	8	Shepherd (1550 Degrees Cent.)
D	Open-Hearth	6	A.S.T.M.
E	Open-Hearth	4	A.S.T.M.
F	Electric Furnace	6–7	A.S.T.M.

The manufacturers rated E as a coarse-grained steel, D and F as fine-grained, and A approximating coarse-grained steel or as closely coarse as commercially practicable. Since the temperature used for the A. S. T. M. test is different from that used for the Shepherd test, the two sets of values cannot be compared. Of the two steels given the Shepherd test, B had the coarser grains. More strictly comparable grain size determinations of the steels are discussed in a later section of this paper.

Preliminary Gradient Tempering of Steel X. It appears to be generally assumed that the decrease in hardness of a quenched high carbon steel increases with the tempering temperature until the critical range is reached. However, the results obtained by a gradient tempering of steel X after quenching in cold water from 950 degrees Cent. (1740 degrees Fahr.) shows a minimum hardness at a temperature well below the critical (Fig. 3). The tempering temperatures ranged from 450 to 800 degrees Cent. (840 to 1470 degrees Fahr.),

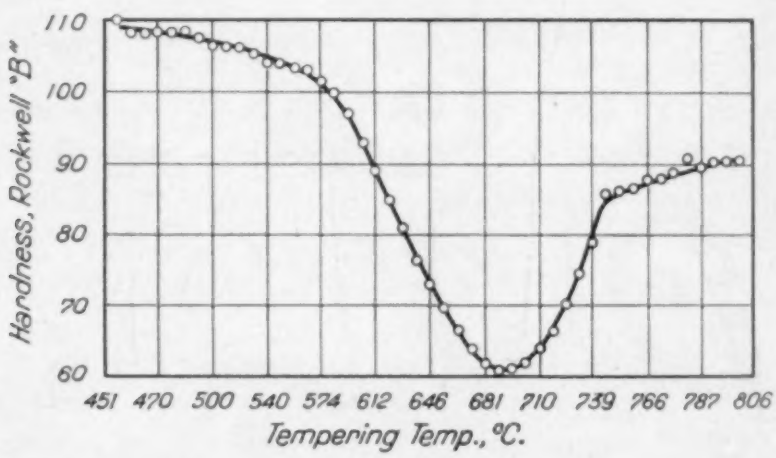


Fig. 3—Hardness Distribution in Steel X, Quenched After 1 Hour at 950 Degrees Cent., Tempered for 48 Hours in Furnace with a Gradient Temperature, and Furnace Cooled.

and the bar was annealed for 48 hours and then allowed to cool slowly in the furnace. The diagram shows a minimum hardness at about 690 degrees Cent., well below the lower critical.

A study of the change in microstructure along the bar revealed some microstructural changes which were not anticipated. Representative photomicrographs observed at a magnification of 2000 diameters are reproduced in Fig. 4. These show the structure formed at 620, 675, 725, and 745 degrees Cent. (1150, 1250, 1340, 1375 degrees Fahr.), respectively. It is apparent that the carbides grow

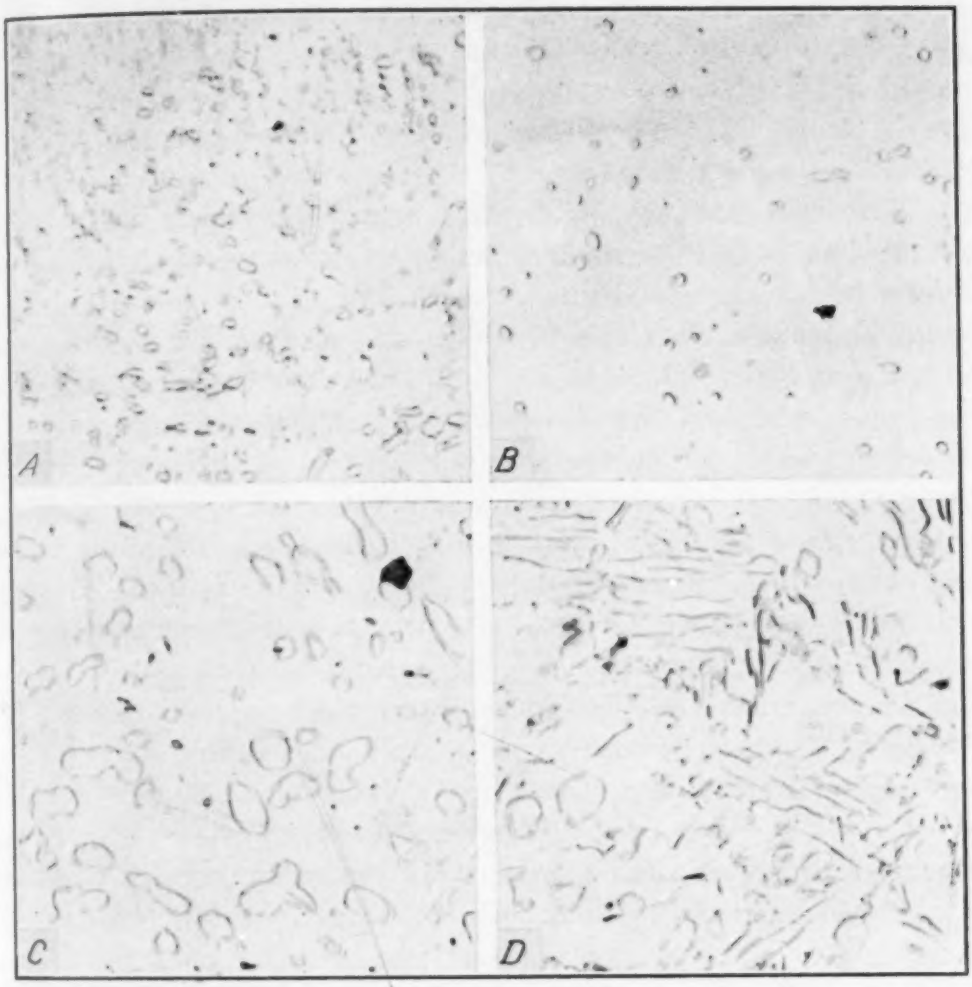


Fig. 4—Microstructures from Steel X After Treatment in Gradient Furnace (Fig. 3). Tempering Treatment: A.—620 Degrees Cent. B.—675 Degrees Cent. C.—725 Degrees Cent. D.—745 Degrees Cent.

larger as the temperature is increased. However, the change in size is not proportional to the temperature since very little change has taken place between 620 and 675 degrees Cent. (1150 and 1250 degrees Fahr.) while a large change is noted between 675 and 725 degrees Cent. (1250 and 1340 degrees Fahr.). Although the carbides have practically the same size at 620 and 675 degrees Cent., more of them are present at the lower temperature. Thus, the decrease in hardness that takes place within this temperature range is partially, if not entirely, due to a reduction in the amount of carbide. It will be shown later that the disappearance of the carbide is caused by its decomposition into iron and graphite. Figs. 4C and 4D clearly show that the A_1 critical point lies between 725 and 745 degrees Cent. (1340 and

1375 degrees Fahr.) and not at the temperature where the minimum point occurs in the hardness curve.

Experimental Procedure

There are several aspects which may be considered in arriving at a decision as to the optimum method of quenching a series of high carbon steels of similar chemical composition, prior to a study of their tempering characteristics. However, it was arbitrarily decided to heat all specimens to 1000 degrees Cent. (1830 degrees Fahr.), maintain for 1 hour and then quench in cold water. The basis of this choice was the conception that the prolonged anneal at so elevated a temperature could be expected to largely eliminate the effects of differences in fabrication history and in final treatment of the steels before they were received by the authors. Details of the results of various heat treatments on the several steels prior to quenching are given in a later section of the paper.

The specimens for hardness determinations and for metallographic study were ⅜-inch diameter and ½-inch long. They were hardened by heating in a closed electric tube furnace for 60 minutes at 1000 degrees Cent. (1830 degrees Fahr.) and quenched in cold water. A series of such specimens was then tempered at 550, 650 or 710 degrees Cent. (1020, 1200, 1310 degrees Fahr.) for periods varying from 30 minutes to 125 hours, followed by water quenching. A different specimen was used for each tempering experiment, so that all samples were heated only once to the tempering temperature and finally quenched. The tempering temperatures were controlled to within ± 0.5 degree Cent. by use of a phototube control unit built in the laboratory. A lead bath was used for the purpose. After cutting transversely, one half of the specimen provided material for hardness data, the other half was used for metallographic examination.

The specimens for determinations of electrical resistivity and coercive force were 5½ inches long and 0.25 ± 0.001 inches in diameter. A softening treatment was first given to the steels in order to permit machining to a uniform ¼-inch diameter. They were then hardened by heating in a salt bath of "carbon lavite" at 1000 degrees Cent. (1830 degrees Fahr.) for 60 minutes and quenched in cold water. The specimens were preheated before immersing in the salt bath in order to militate against cracking. Duplicate specimens were

then tempered at 550, 650 and 710 degrees Cent. (1020, 1200, 1310 degrees Fahr.) for progressively increasing periods, each tempering operation being followed by quenching in cold water. Thus in this series of tests the bars were repeatedly heated to and quenched from one given tempering temperature. This repeated heating and cooling, as distinct from the single treatment for the hardness tests, will later be shown to have significance. The tempering was conducted *in vacuo* in an electric tube furnace with temperature controlled to $\pm$ 2 degrees Cent. The temperature gradient along the bars was less than 1 degree Cent.

The complete sequence of heat treatments was conducted in a manner to render the treatments as strictly comparable as possible for the different steels.

Experimental Results

In this section are presented the data obtained from a study of the effects of tempering the steels at 550, 650 and 710 degrees Cent. (1020, 1200, 1310 degrees Fahr.), after quenching from 1000 degrees Cent. (1830 degrees Fahr.), on electrical resistivity, magnetization coercive force, and Rockwell hardness "B."

The data on resistivity and coercive force are given in Tables III and IV respectively. The values under zero hours indicate the results obtained immediately on quenching and the subsequently recorded values were obtained after successive temperings at the given temperature for ½, 1, 5, 25, 75, and 125 hours, followed by water quenching. Two values are noted for a specific treatment for each steel, and they represent the mean of three observations for each of the duplicate samples tested.

The hardness data obtained on the ½-inch long samples are recorded in Table V, in similar form. However, only one value is given for each specific treatment and this number represents the average of three observed values. A different quenched sample was used for each of the different recorded times at tempering temperature, but no specimens were prepared for examination in the as-quenched state.

A general picture of the effects of time at temperature on the six steels with reference to the three properties considered is obtained by a study of Figs. 5, 6, and 7 which relate to treatments at 550, 650, and 710 degrees Cent. (1020, 1200, 1310 degrees Fahr.) respectively.

Table III

Specific Resistance—10^{-6} Ohms Per Centimeter Cube

Steel	Time at 550 Degrees Cent.—Hours							Time at 650 Degrees Cent.—Hours							Time at 710 Degrees Cent.—Hours						
	0	½	1	5	25	75	125	0	½	1	5	25	75	125	0	½	1	5	25	75	125
A	39.45	22.0	21.65	21.2	20.6	19.1	18.85	40.0	21.2	21.05	20.45	19.5	19.15	18.85	38.85	20.6	20.4	20.35	20.0	19.25	19.05
	39.35	21.9	21.5	21.2	20.45	19.05	18.7	39.9	20.9	20.85	20.4	19.25	18.75	18.8	38.45	21.1	21.2	21.2	20.75	20.2	20.1
B	41.7	26.0	26.1	25.5	25.15	25.0	25.0	43.0	25.7	25.85	25.6	25.1	23.9	23.35	41.7	24.8	24.8	24.8	24.65	24.3	24.6
	41.2	25.55	25.4	25.0	24.5	24.2	24.35	43.5	25.9	26.0	25.65	25.3	23.7	23.6	41.0	25.1	25.4	25.2	25.05	24.95	25.1
C	40.2	22.8	22.0	21.6	21.2	20.8	20.65	41.5	21.8	21.7	21.25	20.6	19.65	19.25	40.6	21.7	21.6	21.55	21.4	21.2	21.5
	40.7	22.75	22.25	21.7	21.5	21.25	21.0	40.6	21.8	21.8	21.4	20.75	19.4	19.25	41.0	22.65	23.0	22.8	22.7	22.8	23.25
D	37.6	22.15	21.95	21.6	21.15	20.15	20.2	37.2	20.2	20.15	19.7	19.2	19.05	18.8	37.8	20.3	20.2	20.5	20.15	19.9	19.8
	37.0	20.9	20.65	20.4	20.05	19.35	19.0	37.6	....	.....	....	....	.....	....	34.2	19.2	19.3	19.1	18.9	18.7	18.75
E	35.9	20.25	20.1	19.9	19.6	19.1	19.15	35.9	20.0	19.85	19.45	19.3	18.8	18.8	36.3	20.3	20.45	20.2	20.0	19.75	20.0
	35.7	20.65	20.6	20.25	20.15	19.9	19.7	35.85	19.65	19.55	19.2	19.05	18.4	18.0	34.9	20.1	20.1	19.7	19.8	19.45	19.85
F	40.1	23.75	23.75	23.75	23.0	21.75	21.7	42.1	24.05	24.2	23.9	23.1	23.0	23.35	39.9	22.4	22.5	22.4	21.95	21.45	21.6
	39.1	23.1	23.25	22.7	22.25	21.3	21.0	41.6	23.8	24.1	23.6	22.8	22.7	22.8	36.9	21.2	21.05	21.0	20.95	20.4	20.55

Table IV

Magnetization-Coercive Force—Oersteds

Steel	Time at 550 Degrees Cent.—Hours					Time at 650 Degrees Cent.—Hours					Time at 710 Degrees Cent.—Hours				
	0	½	1	5	25	75	125	0	½	1	5	25	75	125	
A	37.9	21.2	20.3	19.2	17.8	14.2	11.65	39.8	17.1	16.7	14.0	8.95	5.96	4.95	
	37.8	20.9	20.3	19.15	17.5	14.2	11.7	39.7	17.3	16.4	14.0	9.2	6.05	5.1	
B	36.4	20.9	20.3	19.35	17.4	16.4	15.65	38.2	16.8	16.35	13.9	11.15	6.7	4.85	
	36.5	20.9	20.2	19.3	17.6	16.2	15.6	37.9	17.0	16.1	14.15	11.3	6.75	4.9	
C	38.3	21.8	21.0	19.6	18.1	16.55	15.7	38.8	17.3	16.7	14.2	10.6	6.3	4.85	
	37.7	21.4	20.7	19.6	17.7	16.3	15.5	38.7	17.4	16.5	14.4	10.85	6.45	5.1	
D	38.8	19.8	18.9	17.95	16.8	14.35	12.2	38.8	15.8	15.1	12.95	9.15	6.15	4.85	
	38.9	19.6	18.9	18.0	16.6	14.3	12.1	40.3	15.0	14.9	12.9	9.25	6.0	5.05	
E	38.2	19.4	18.6	17.45	15.9	14.7	14.05	39.4	15.3	14.7	12.95	10.9	8.7	6.45	
	37.7	19.2	18.5	17.35	15.7	14.5	14.0	39.5	15.5	14.8	13.1	10.85	8.55	6.5	
F	38.6	20.7	19.8	18.6	16.6	13.1	10.8	39.85	16.4	15.7	13.0	8.35	5.05	4.1	
	38.3	20.4	19.8	18.65	16.4	13.0	10.7	40.1	16.5	15.8	13.1	8.0	5.05	4.3	

Steel	0	½	1	5	25	75	125	0	½	1	5	25	75	125	
	38.2	14.25	13.3	11.5	8.9	6.4	5.35								
	36.0	14.05	13.4	11.5	9.2	6.8	5.4								
	38.7	13.9	13.1	11.4	9.7	8.2	7.5								
	35.25	13.7	13.1	11.1	9.4	8.25	7.5								
	38.7	14.2	13.25	11.7	9.95	8.45	7.75								
	36.6	14.3	13.7	11.8	9.85	8.35	7.75								
	39.75	13.25	12.6	11.0	9.15	7.25	5.85								
	36.6	13.0	12.5	10.55	8.95	7.15	6.05								
	38.25	12.4	12.0	10.2	8.75	7.65	6.7								
	36.95	12.55	12.0	10.3	8.7	7.4	6.9								
	39.3	13.55	12.8	11.3	8.85	6.0	4.8								
	37.0	13.7	13.2	11.0	9.1	5.9	4.75								

Table V

Rockwell "B" Hardness (Hardness Specimens)

Steel	Time at 550 Degrees Cent.—Hours							Time at 650 Degrees Cent.—Hours							Time at 710 Degrees Cent.—Hours						
	½	1	5	25	75	125		½	1	5	25	75	125		½	1	5	25	75	125	
A	109.7	109.3	107.5	106.1	101.1	97.6		103.8	102.7	99.6	88.0	80.2	78.8		99.2	97.7	94.3	90.2	85.6	83.0	
B	109.9	109.0	108.0	106.4	102.7	102.4		104.2	103.5	99.7	95.7	93.1	90.9		99.4	97.7	94.2	90.3	87.6	87.4	
C	110.2	109.4	108.1	105.8	103.0	101.8		104.4	103.1	100.2	95.8	93.8	93.0		99.5	99.3	93.2	90.8	88.4	89.3	
D	109.3	108.7	107.9	105.3	101.2	98.7		102.9	102.5	98.2	89.5	78.4	74.7		99.0	94.7	92.0	88.7	83.2	83.9	
E	109.0	108.4	107.2	103.4	100.3	98.2		102.8	101.4	97.2	92.2	89.8	88.8		98.1	99.4	91.5	90.4	82.4	82.1	
F	109.4	108.9	106.9	103.9	97.7	93.7		103.2	102.7	99.6	93.9	77.3	71.5		98.0	96.6	93.4	88.6	86.7	88.0	

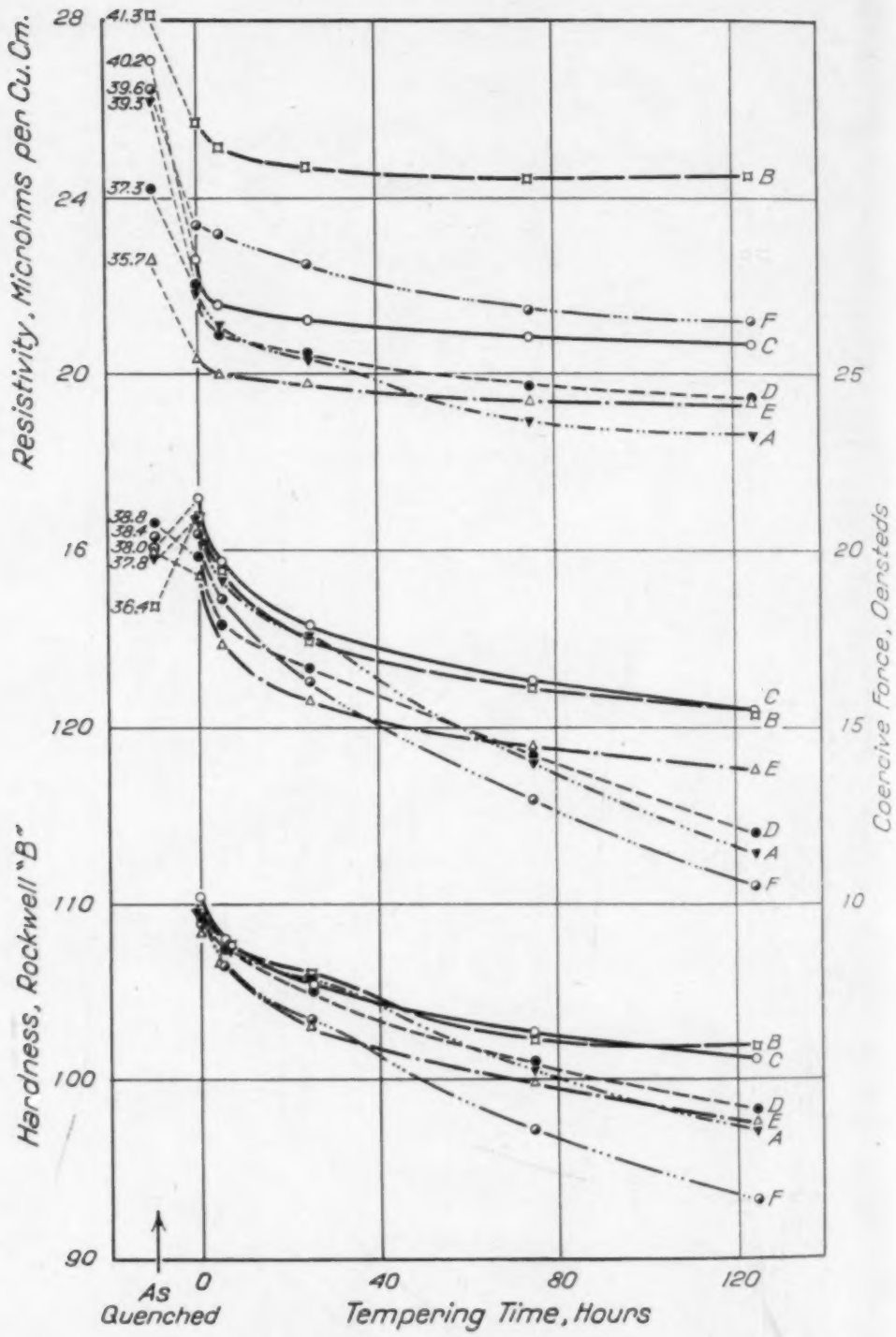


Fig. 5—Effect of Time of Tempering at 550 Degrees Cent. on Resistivity, Coercive Force, and Hardness.

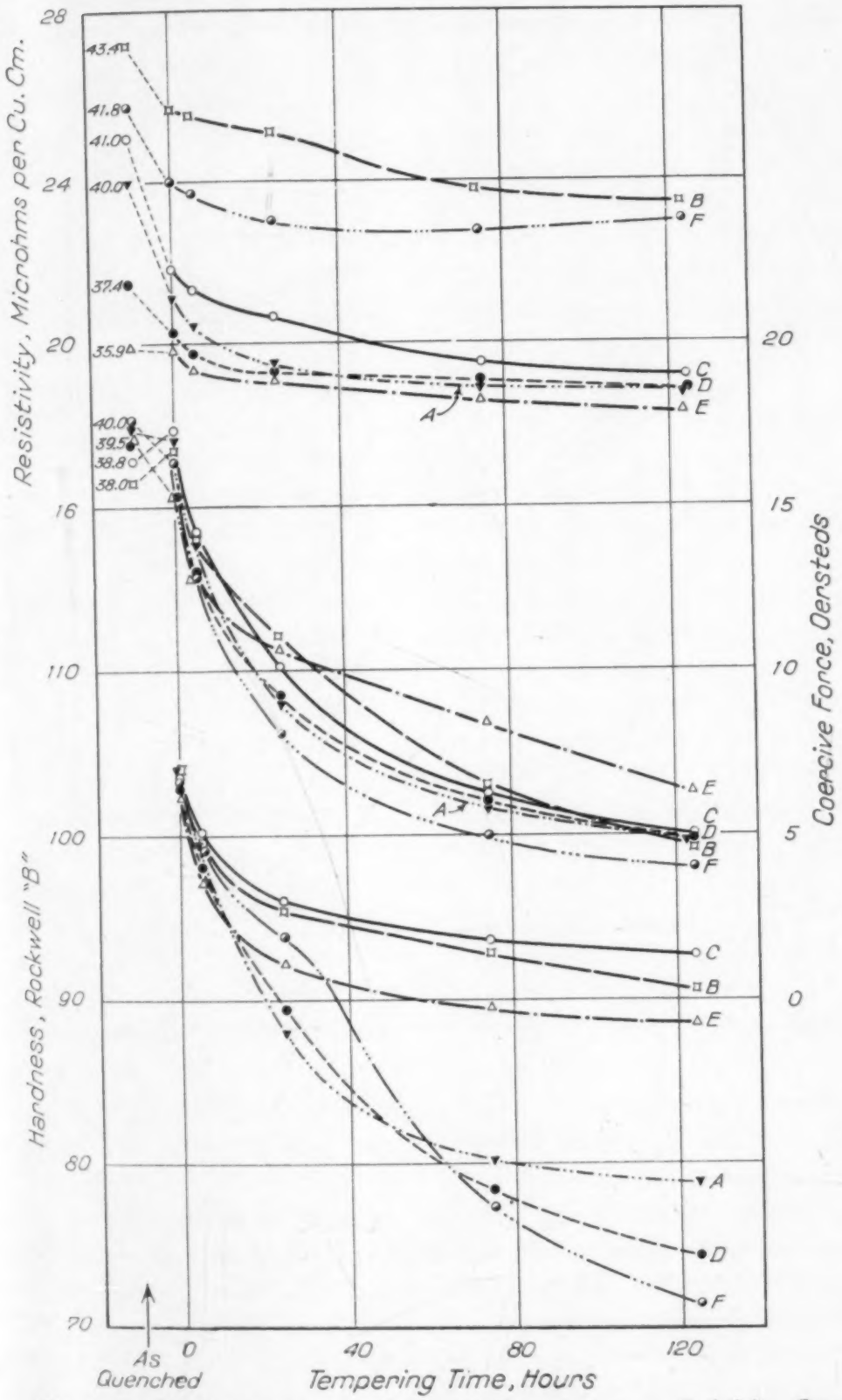


Fig. 6—Effect of Time of Tempering at 650 Degrees Cent. on Resistivity, Coercive Force, and Hardness.

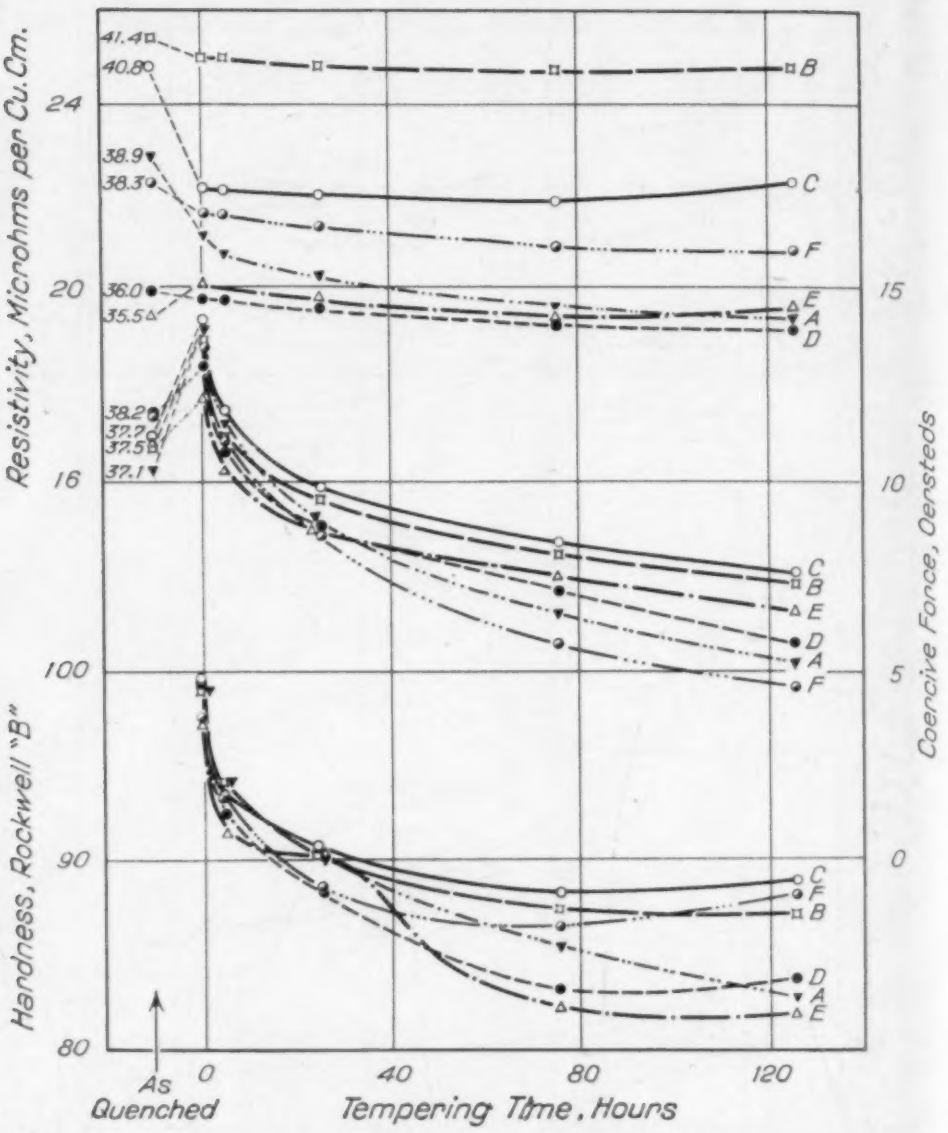


Fig. 7—Effect of Time of Tempering at 710 Degrees Cent. on Resistivity, Coercive Force, and Hardness.

Average values for the two specimens of each steel have been plotted, with the data for 1 hour temper omitted, in order to avoid congestion at this part of the graph.

Electrical Resistivity. Despite the approximate similarity in the chemical composition of the six steels, a marked difference was noted in the electrical resistance of the quenched samples. The resistivity of Steel B exhibited a maximum in each of the three series, while Steel E shows the lowest value. The general order indicates that silicon is playing a prominent part. B has the most silicon while E and D have the least. B and E have similar carbon contents.

Since E and D have about the same manganese and silicon contents, it might be considered that E should have the greater resistivity by virtue of its higher carbon. The experimental data show that the reverse case obtains. Possibly the presence of copper in Steel E lowers the resistivity by virtue of its tendency to reject carbon from solid solution. It may also be noted that the copper-bearing Steel A exhibits low specific resistance on quenching. No data are available for the copper content of the other steels. The relatively high value found in Steel F despite the low carbon content may be attributed to the higher nickel content and its effect on slowing the rate of transformation on cooling.

The resistivity of the tempered steels also appears to be influenced by their silicon content, and for the 710 degrees Cent. (1310 degrees Fahr.) tempering tests the steels have the same order for their resistances as they do for the silicon contents. As the duration of the tempering treatment increases the resistances of A, D, and E approach each other. B, C, and F on the other hand tend to deviate. The latter three steels were made in an electric furnace while the former were produced in an open-hearth furnace.

At 550 and 650 degrees Cent. (1020 and 1200 degrees Fahr.) the progressive change in resistivity of the steels does not bear as close a relationship to the silicon content as it did at 710 degrees Cent. (1310 degrees Fahr.). Thus at 550 degrees Cent. (1020 degrees Fahr.) D has much larger resistance than E after short-time tempering, but the values converge as the tempering time is increased. After 125 hours they are practically identical. The difference may be due to the slower precipitation of carbon from alpha iron in D.

The reason for Steel F having a greater resistance than C after tempering at 550 or 650 degrees Cent. (1020 or 1200 degrees Fahr.) may be again accounted for by the rather high nickel content of the former. It is of interest to point out that the electric furnace steels have the highest values, and the open-hearth steels the lowest values for resistivity after tempering at each of the three temperatures investigated.

It is well known that many fundamental reactions follow an exponential law with respect to time. In such cases a semi-logarithmic plot results in the data falling on a straight line. A plot of all resistivity data against log time is presented in Fig. 8 and the result is of particular interest.

On tempering at 550 and 710 degrees Cent. (1020 and 1310 degrees Fahr.) Steels B, C, and E exhibit the linear relation over practically the whole 125 hours of treatment. In the other three steels the rate of decrease in resistivity is greater than would be predicted from the slope of the linear relationship holding up to about 25 hours. This latter fact indicates that some other important fac-

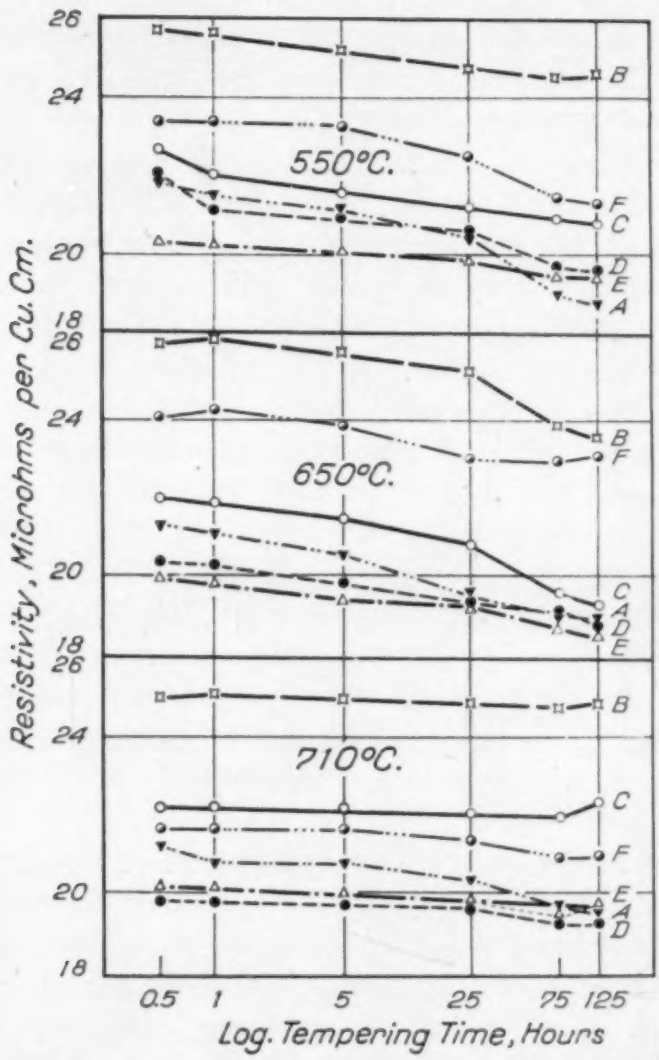


Fig. 8—Semilogarithmic Plot Showing Relation Between Resistivity and Time of Tempering at 550, 650 and 710 Degrees Cent.

tor, not present to any extent in the three former steels, begins to play an important rôle after the first few hours of tempering. At 710 degrees Cent. (1310 degrees Fahr.) the manifestation of this new factor is delayed to a later period than in the 550 degrees Cent.

(1020 degrees Fahr.) tests. None of the steels show the persistence of the linear relation at 650 degrees Cent. (1200 degrees Fahr.) for the entire 125 hours.

Fig. 9 provides a complete résumé of the resistivity data obtained in all the tempering tests, in relation to the three quenched resistivities obtained from each of the steels. The diagram clearly shows how the steels with the highest resistance after hardening are also the highest after tempering. Steels B and F, which had a much higher resistivity prior to the 650 degrees Cent. (1200 degrees Fahr.) temper than before the other treatments, show that the hardening treatment may have a profound effect upon the resistance found after tempering. With the exception of F, all of the steels had a lower resistivity after the 650 degrees Cent. (1200 degrees Fahr.) treatment than after the other two tempering treatments. For short tempering times, B reacted similarly to F. In all steels the length of tempering time has less effect at 710 degrees Cent. (1310 degrees Fahr.) than at either of the other two temperatures.

On account of the fact that wide divergence was obtained in the quenched electrical resistivity of different samples of the same steel, a plot was prepared to illustrate the decrease in resistivity with temperature and time without respect to absolute values. The results are shown in Fig. 10, and the general uniformity in the nature of the behavior of the steels is at once evident. Similar data for coercive force are also included in this figure to afford ready comparison.

A study of the data will reveal that the steels with the higher carbon contents decrease in resistivity to a greater extent upon tempering. They have available the most carbon for retention in the quenched martensite, and this increases the resistance. On tempering they reject more carbon from solution in the iron and consequently exhibit a greater decrease in resistivity. The relatively small change in Steel E has been noted previously.

Magnetization Coercive Force. Data similar to those presented for resistivity are also included for coercive force, in Figs. 5, 6, and 7.

Steels A, B, and E will be later shown to have the coarsest grain size and it may be noted that they have the lower coercive force in the quenched state. This correlates with reaction rates, dependent on grain size, since small austenite grains have a greater tendency to transform than coarse grains. Thus the greatest

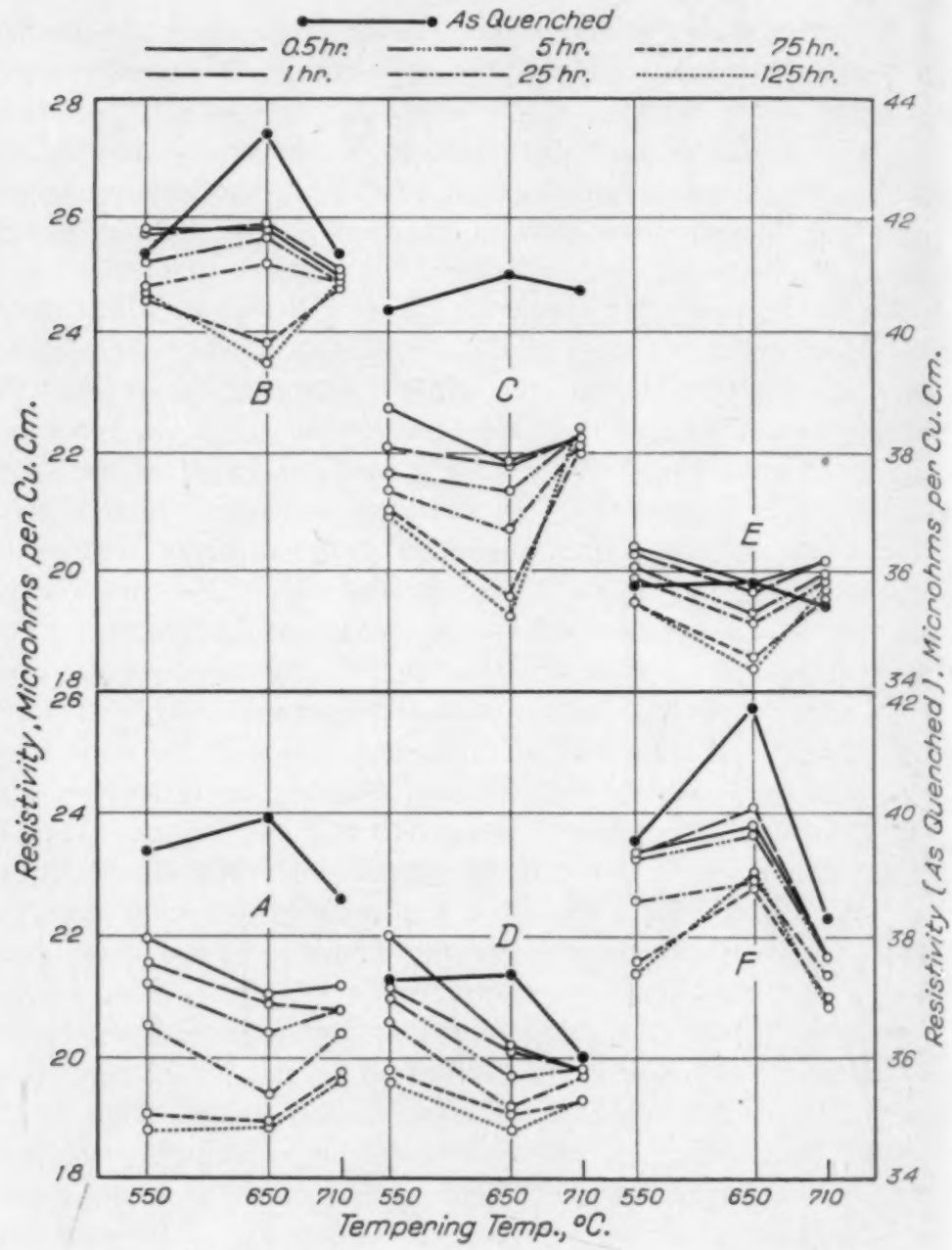


Fig. 9—Effect of Temperature and Time of Tempering on Resistivity.

amount of carbide might be expected to be ejected from the solid solution of a fine-grained steel during quenching, with resultant higher coercive force. After tempering for 5 hours or less at any of the three temperatures, A, B, and C have the highest values for coercive force and these are the steels having the highest carbon content, and hence the largest amount of carbide. The low values observed on Steel E at short tempering times do not correlate with

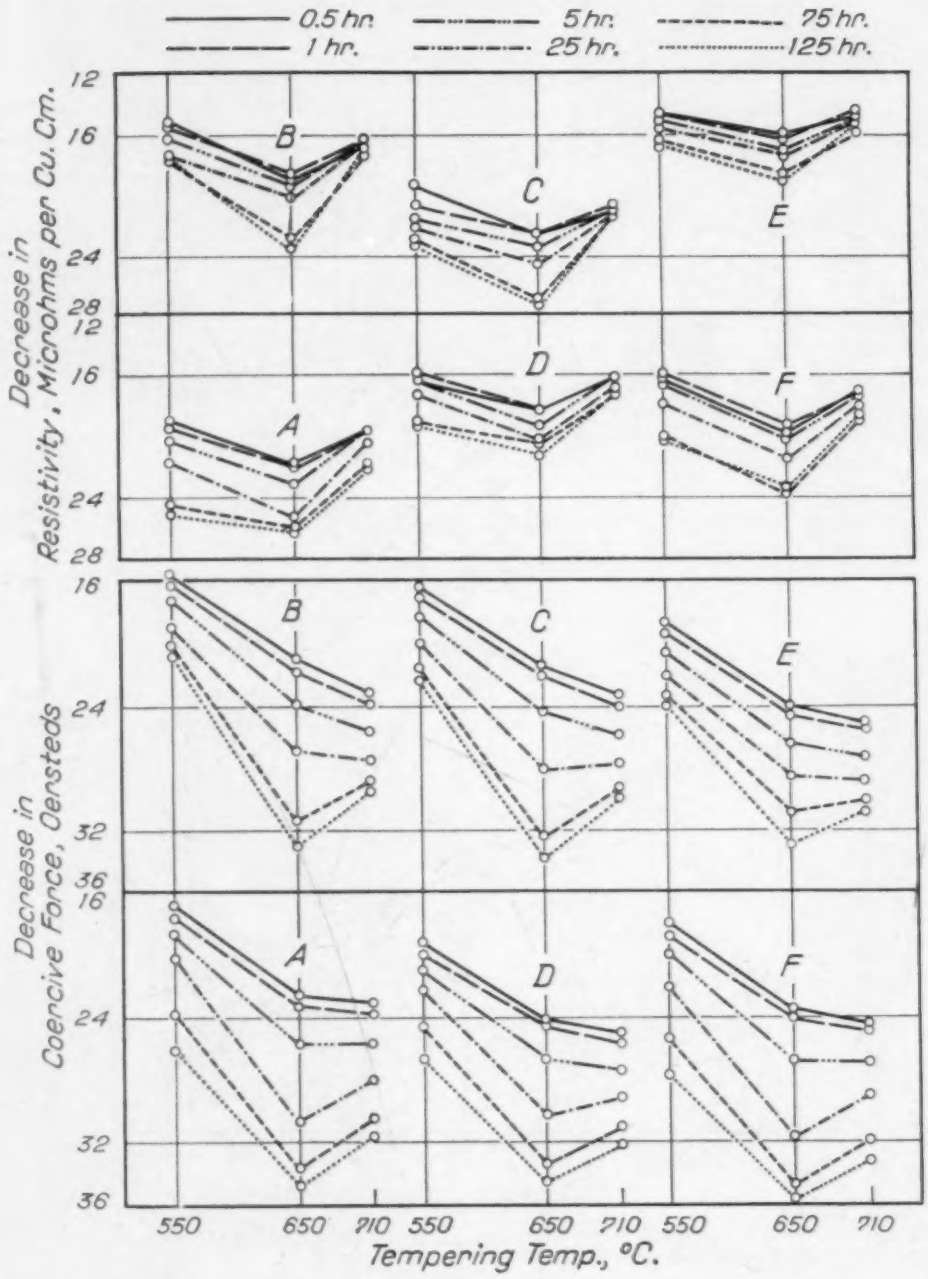


Fig. 10—Decrease in Resistivity and Coercive Force from Value for Quenched Steel, in Relation to Temperature and Time of Tempering.

the carbon, as was the case in considering resistivity. Again the presence of copper may influence the data.

After 25 hours at 550 and 710 degrees Cent. (1020 and 1310 degrees Fahr.) there is a distinct separation of the steels into two groups. One group, consisting of Steels B, C, and E, has the high values, and the curves appear to approximate to linearity. At 550

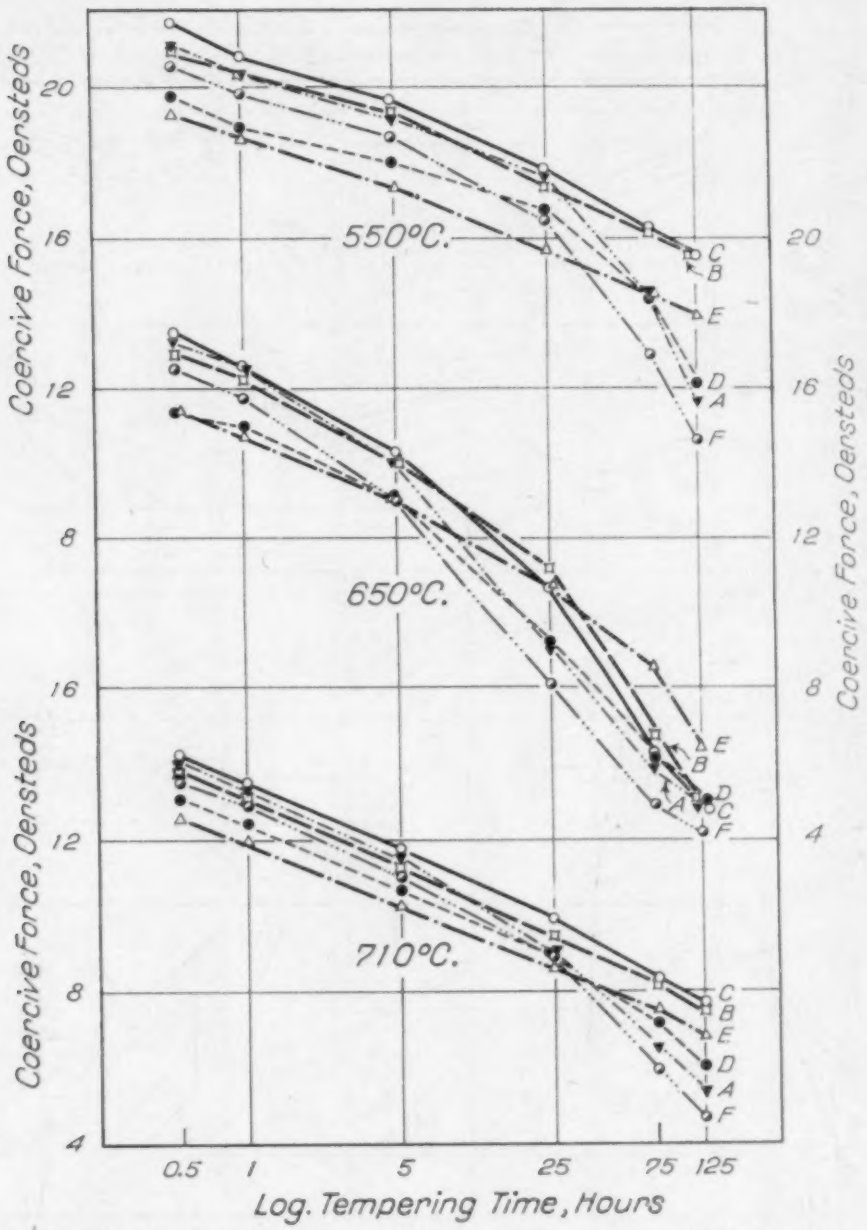


Fig. 11—Semi-logarithmic Plot Showing Relation Between Coercive Force and Time of Tempering at 550, 650 and 710 Degrees Cent.

degrees Cent. (1020 degrees Fahr.) the second group, Steels A, D, and F, shows a continuation of the rapid decrease in coercive force even after 125 hours. At 710 degrees Cent. (1310 degrees Fahr.) all three steels are markedly lower than those in the first group but the rate of decrease of all steels between 75 and 125 hours is approximately the same. The peculiar behavior of Steel E is common to all tempering temperatures.

Again at 550 and 710 degrees Cent. (1020 and 1310 degrees

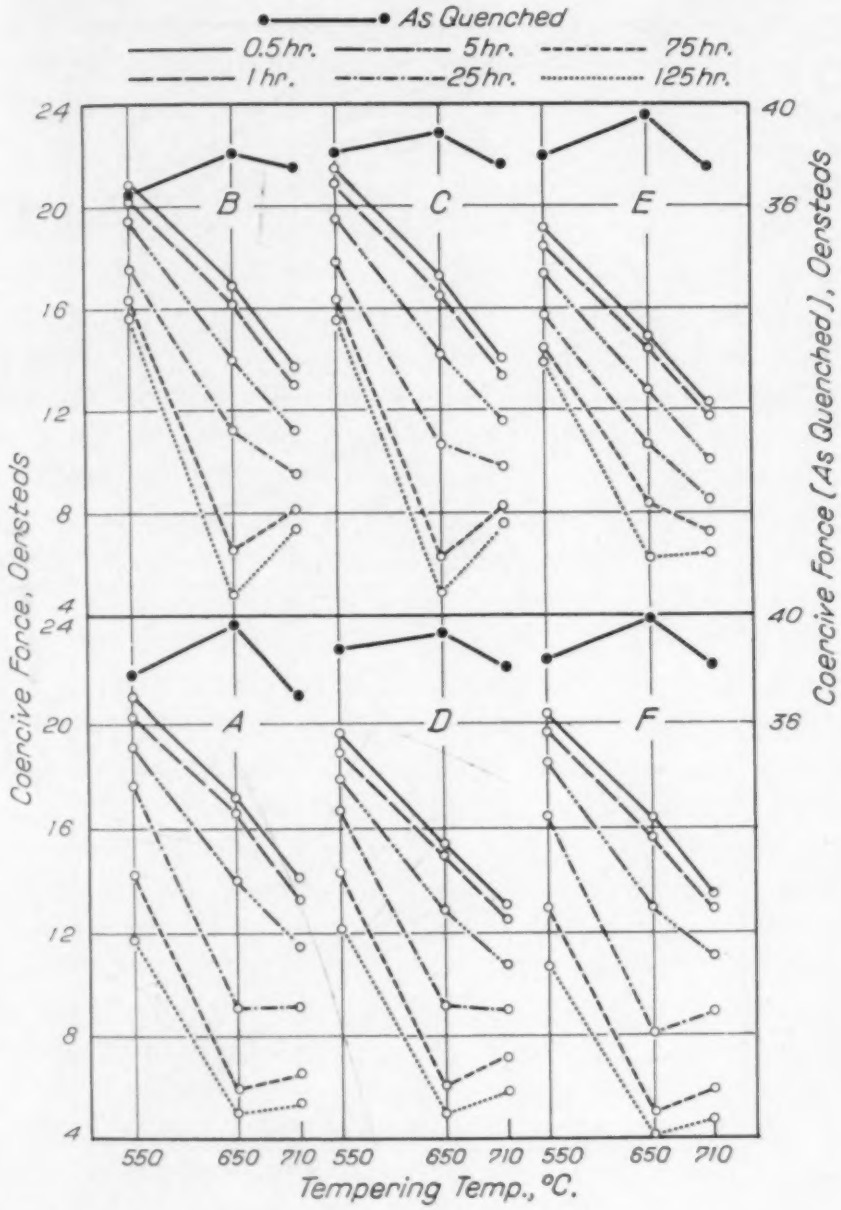


Fig. 12—Effect of Temperature and Time of Tempering on Coercive Force.

Fahr.) Steels B and C which behave similarly on long tempering and have the greatest coercive force, contain the most carbon and silicon and have the lowest manganese. No grouping of the steels appeared possible as a result of the data obtained from tempering at 650 degrees Cent. (1200 degrees Fahr.).

A semi-logarithmic plot reveals in a more striking manner the differences existing between the coercive forces of the various steels after a given tempering treatment. Since decrease in coercive force

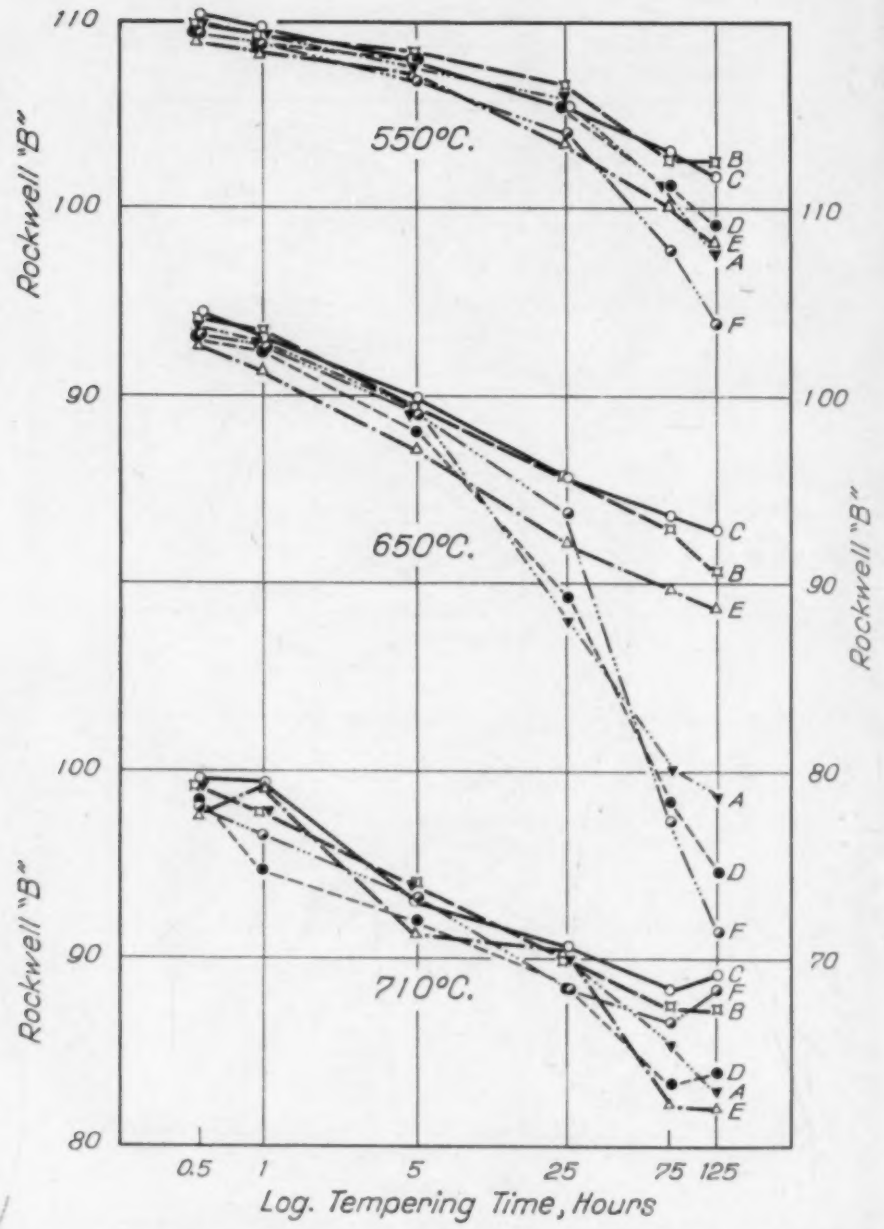


Fig. 13—Semi-logarithmic Plot Showing Relation Between Hardness and Time of Tempering at 550, 650 and 710 Degrees Cent.

is probably a function of carbide particle size, and this in turn is controlled by rate of diffusion, one may have anticipated a linear function for the plot given in Fig. 11. In many instances, at least within certain time limits of tempering, this linearity is remarkably well demonstrated by the data obtained.

At 550 degrees Cent. (1020 degrees Fahr.) the curves of B, C, and E are practically linear for the entire test of 125 hours. The other three steels exhibit an approximate linear relationship for the

first few hours of tempering and then begin to fall off rapidly. This indicates the introduction of a new factor which is profoundly modifying the coercive force as the time of tempering is prolonged. At 650 degrees Cent. (1200 degrees Fahr.) all steels with the exception of E exhibit a tendency for the coercive force to fall off more rapidly than might be anticipated from considerations of diffusion and aggregation alone. The curve for Steel E shows only a very slight dip even up to 75 hours of tempering. At 710 degrees Cent. (1310 degrees Fahr.) a grouping similar to that observed in the data for 550 degrees Cent. (1020 degrees Fahr.) may be noted.

It is of interest to direct attention to the fact that Steels B and C, at all temperatures, behave as if they were the same steel. Their reaction was also similar in the resistivity tests. Steels A and F are likewise not very different in behavior.

In Fig. 12 is shown the general relationship between coercive force and temperature. For short periods of tempering the fall in coercive force is proportional to temperature, but after 5 hours the coercive force decreases much more rapidly at 650 than at 550 or 710 degrees Cent. (1200, 1020 or 1310 degrees Fahr.). It may be noted, however, that the rate of change with time at 550 and 710 degrees Cent. is about constant for each of the steels. The previously mentioned grouping of the steels into B, C, E, and A, D, and F is also evident in Fig. 12. Note the relatively small effect of time at 550 and 710 degrees Cent. (1020 and 1310 degrees Fahr.) in the upper group (Steels B, C, E) as compared with the much larger effect in the lower groups (Steels A, D, F). Reference should also be made to Fig. 10 where the decrease in coercive force, below the value obtained on quenching, is plotted against the temperature in the form of a family of curves representing the series of tempering times. This diagram emphasizes the self grouping of the steels.

Rockwell "B" Hardness. The plot of the Rockwell hardness test data of the ½-inch samples resulting from tempering for periods up to 125 hours is given in Figs. 5, 6, and 7. At 550 degrees Cent. (1020 degrees Fahr.) the results of tempering on the fall in hardness correlates quite well with the data for coercive force. Steel E exhibits the major difference but it must be remembered that a separate specimen was used for each heat treatment in the hardness tests, while the coercive force samples were subjected to all the treatments at a given temperature. This means that the former

were heated to temperature and quenched only once, while the latter were repeatedly heated and quenched. This repeated heating and quenching appears to hasten certain reactions.

At 650 degrees Cent. (1200 degrees Fahr.) the above mentioned correlation of data completely disappears but the aforementioned groupings into series B, C, E and A, D, F is remarkably well shown. At 710 degrees Cent. (1310 degrees Fahr.) the same tendency to group is evident with the exception that at 75 to 125 hours Steels E and F interchange their group selection.

The semi-logarithmic plot of the hardness data (Fig. 13) indicates a fair approximation to linearity in nearly all the tests up to at least 5 hours tempering time. Evidently some scatter of the data from the 710 degrees Cent. (1310 degrees Fahr.) treatment leads to the irregular type of curves noted in the plot. At 550 degrees Cent. (1020 degrees Fahr.) all the curves bend downward beyond 5 or 25 hours but Steels B and C exhibit the greatest resistance to softening and thus correlate with the data for coercive force. While the grouping of the steels is well emphasized by the plot for 650 degrees Cent. (1200 degrees Fahr.) the interesting feature is the linearity exhibited by Steels B, C, and E.

At 710 degrees Cent. (1310 degrees Fahr.), despite the scatter of the data, a general linear relationship is evident in three of the steels, while the other three show a marked but limited drop in hardness toward the end of the full tempering period. The interchange of E and F in the grouping has been noted previously.

The profound difference in reaction of the steels to softening on tempering is probably most clearly shown by plotting hardness against tempering temperature for each of the six periods—from ½ hour to 125 hours—used in the investigation. The natural arrangement into groups B, C, E and A, D, F is at once evident from a glance at Fig. 14. In the first group the relation between hardness and temperature is approximately linear irrespective of the particular time selected for consideration. However, in the latter group this relationship holds only for the first few hours. Therefore the decrease in hardness at 650 degrees Cent. (1200 degrees Fahr.) occurs at a much greater rate than at either of the other two temperatures. This profound difference is not evident in similar plots relating to electrical resistivity or to coercive force.

Metallographic Study of the Alloys

The study of the structural changes occurring in the alloys, as a result of tempering for various lengths of time up to 125 hours at 550, 650 and 710 degrees Cent. (1020, 1200 and 1310 degrees Fahr.), was conducted on that half of the ½-inch samples which

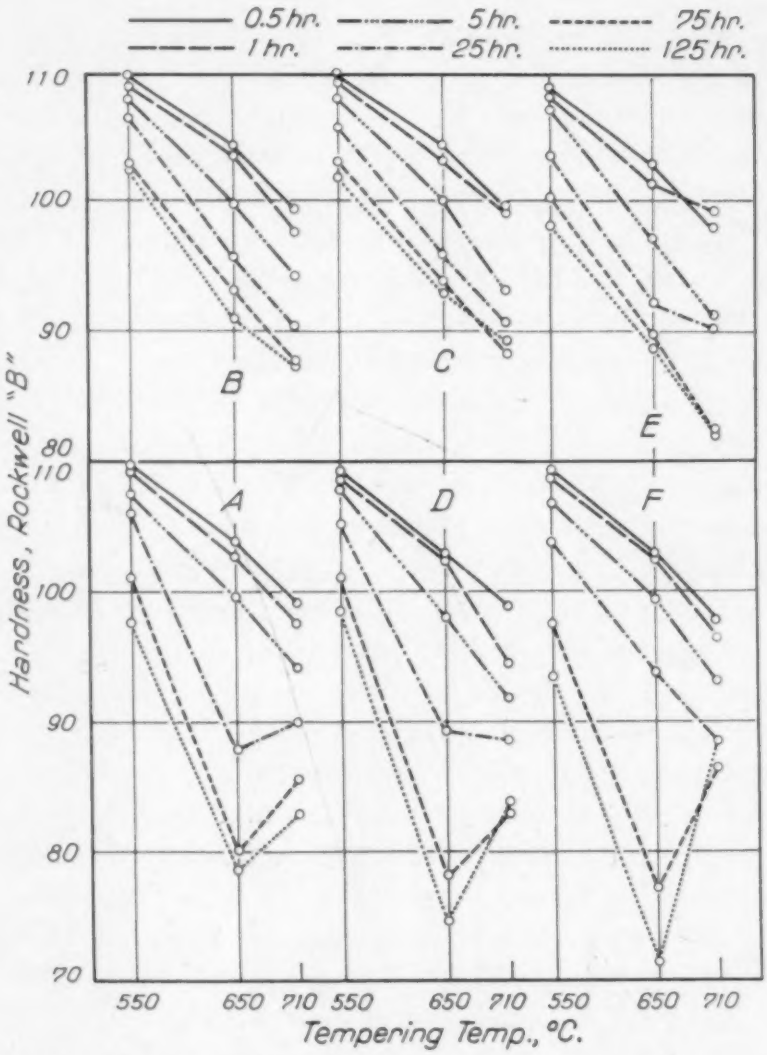


Fig. 14—Effect of Temperature and Time of Tempering on Hardness.

was not subjected to the hardness determinations. Some discussion will also be given to samples cut from resistivity-coercive force samples after the final 125 hours of tempering.

It has been shown in an early section of the paper that apparent anomalous results were obtained for hardness values and micro-

structure during a study of the temperature gradient bar (Steel X) annealed or tempered for 48 hours. Attention has also been directed to the detailed differences observed in resistivity, in coercive force, and in Rockwell hardness, during the study of the effects of tempering from ½ hour to 125 hours at the three selected test temperatures.

The essential purpose of this metallographic study is to obtain some rational explanation for the differences recorded in these steels of "similar" chemical composition. Furthermore, at the risk of apparently overpresenting microstructural records of the work, it has been deemed particularly desirable to furnish as complete a record as practicable illustrating the metallography of typical alloys throughout the history of the heat treatments.

This point is emphasized since it is believed that the present work represents the first published records dealing with a detailed study of the comparison between the reactions, of a series of similar carbon steels, to prolonged tempering treatments. For work of this nature the maximum resolution obtainable by the microscope is frequently inadequate for the purpose of following the mechanism of the reactions. However, with careful attention to specimen preparation technique in grinding, polishing and etching, it is believed that the metallographic records presented in this paper, at a magnification of 2000 diameters, adequately illustrate the profound difference in the reaction of the six steels to tempering.

Lucas (34) has shown by means of photomicrographs some of the changes that take place in the microstructure of a quenched eutectoid carbon steel when it is tempered at various temperatures. He, however, only considered one steel and failed to take into account the effect of time at temperature upon the microstructure.

The first nine plates (Figs. 15 to 23) are arranged in a series of three groups and relate to metallographic observations taken from steels tempered at 550, 650 and 710 degrees Cent. (1020, 1200 and 1310 degrees Fahr.). Each group, representing changes for one of the tempering temperatures, consists of three plates, which in sequence provide the following data:

1. Effect of time of tempering on Steel B (typical of group B, C, E)
2. Effect of time of tempering on Steel F (typical of group A, D, F)
3. Comparison of the resulting structures obtained from all six of the steels after tempering for 125 hours.

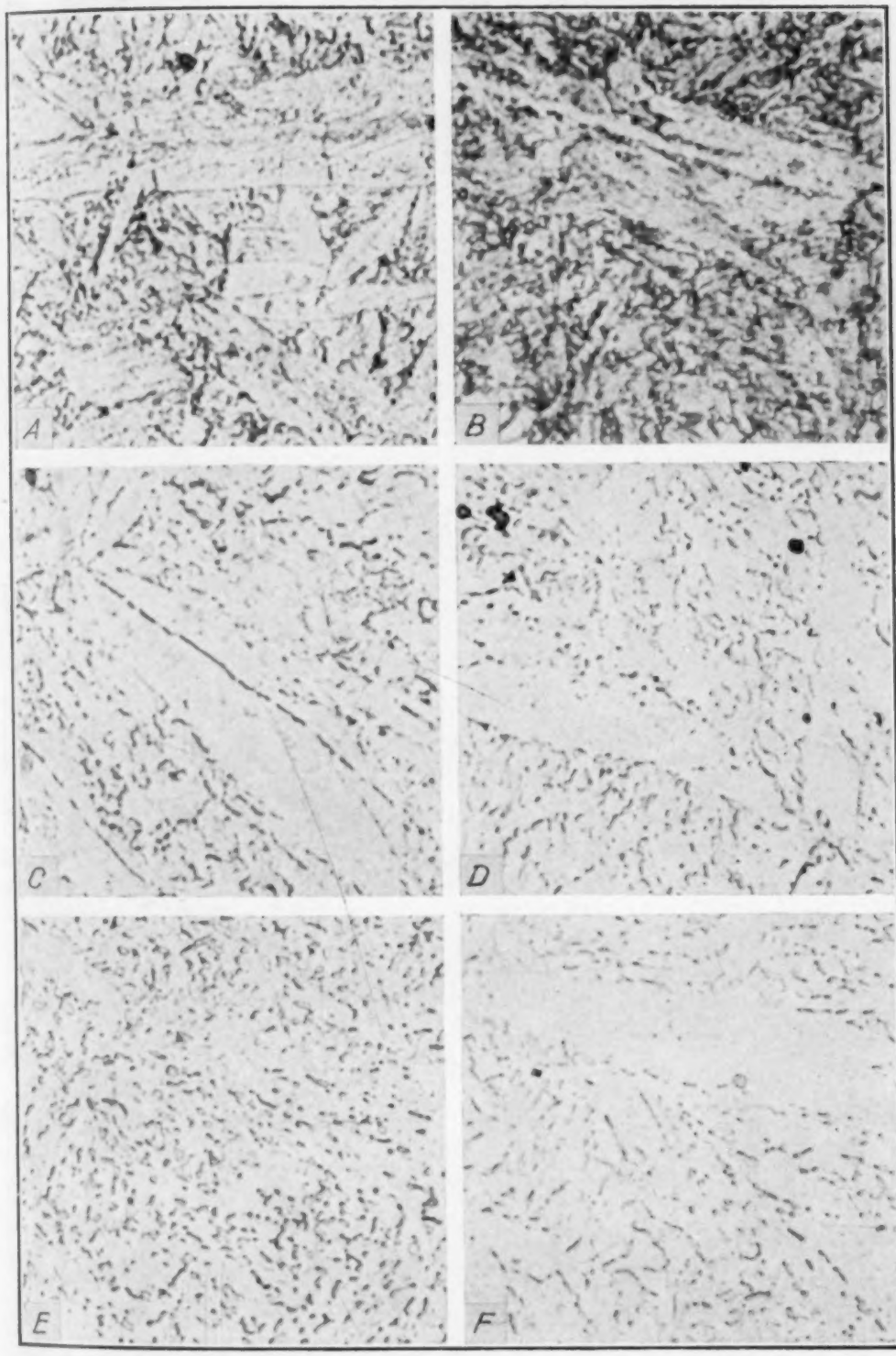


Fig. 15—Photomicrographs of Steel B, Tempered at 550 Degrees Cent.
A.—½ Hour (Light Etch) B.—½ Hour (Deeper Etch) C.—5 Hours
D.—25 Hours E.—75 Hours F.—125 Hours
Picric Acid Etch. × 2000.

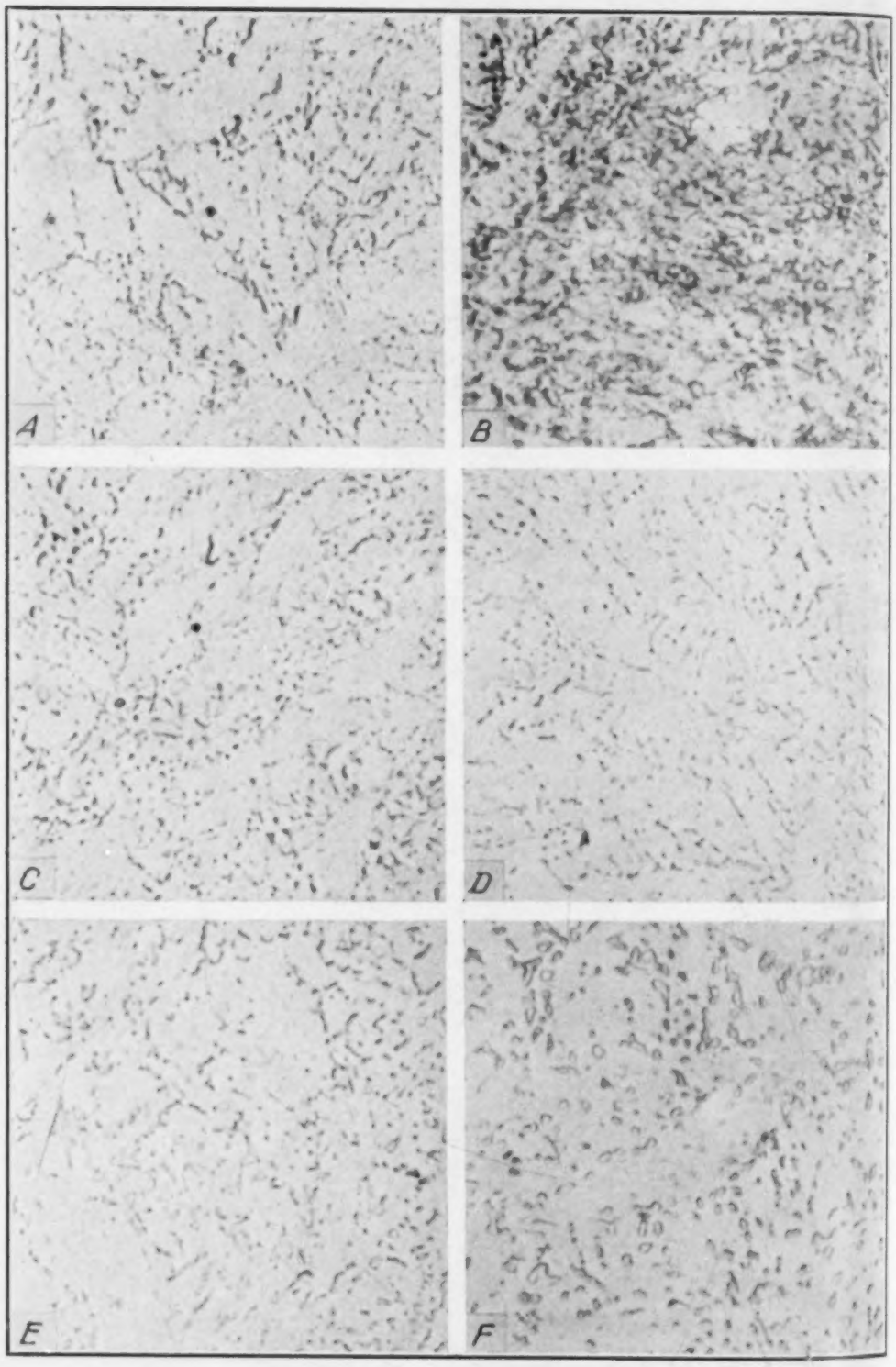


Fig. 16—Photomicrographs of Steel F, Tempered at 550 Degrees Cent.
A.—½ Hour (Light Etch) B.—½ Hour (Deeper Etch) C.—5 Hours
D.—25 Hours E.—75 Hours F.—125 Hours
Picric Acid Etch. × 2000.

All the photomicrographs were taken at a magnification of 2000 diameters with the exception of the unetched samples used to indicate the distribution of graphite. These were recorded at 200 diameters. Metallographic examination showed that similar heat treatments could produce markedly different results in the microstructure of the steels of approximately "similar" chemical analyses. The results of increased tempering time at 550 degrees Cent. (1020 degrees Fahr.) for Steel B and for Steel F in Figs. 15 and 16 illustrate how the martensite needles and hence the pretreatment of the steel control the distribution of the carbides in the subsequently tempered steel. The end product of all the steels, after tempering 125 hours, indicated that the rate of coalescence of the carbides was slowest in B and most rapid in F (Fig. 17). Steel B had the largest austenitic grain size of any of the steels, and F had the smallest. Although, as will be shown later, the fracture of Steel E, after hardening, rated it as a coarse-grained steel, its microstructure was more like that of F, fine grain, than B which had coarse grains. This may be the cause of its tempered structure being different from that of its group (Group B, C, E).

An increase in tempering temperature produces a corresponding increase in the size of the carbides. However, the difference in behavior of what the authors have conveniently considered as the two groups of steels, is at once evident by comparison of the progress of the change in structures of Steels B and F (Figs. 18 and 19) at 650 degrees Cent. (1200 degrees Fahr.). In Steel B evidence of the influence of the original martensite needles on the distribution of the carbides may be noted even after 125 hours at temperature, and the carbides precipitated on the midrib of a needle appear to have the maximum resistance to spheroidization. While the carbides increase in size many develop in an elongated form. In Steel F the influence of the orientation of the original martensite is not easy to follow and after 75 hours all the carbides are spheroidal and have a general random orientation.

A profound difference in the structures is observed in the decrease in the amount of carbide in Steel F as tempering progresses. This phenomenon must be ascribed to graphitization. Again the behavior of the two groups of steels on tempering at 650 degrees Cent. for 125 hours is illustrated in Fig. 20, where the correlation is strikingly evident, both as regards the decrease in the amount of spheroidized carbide and the nature of its distribution. Where

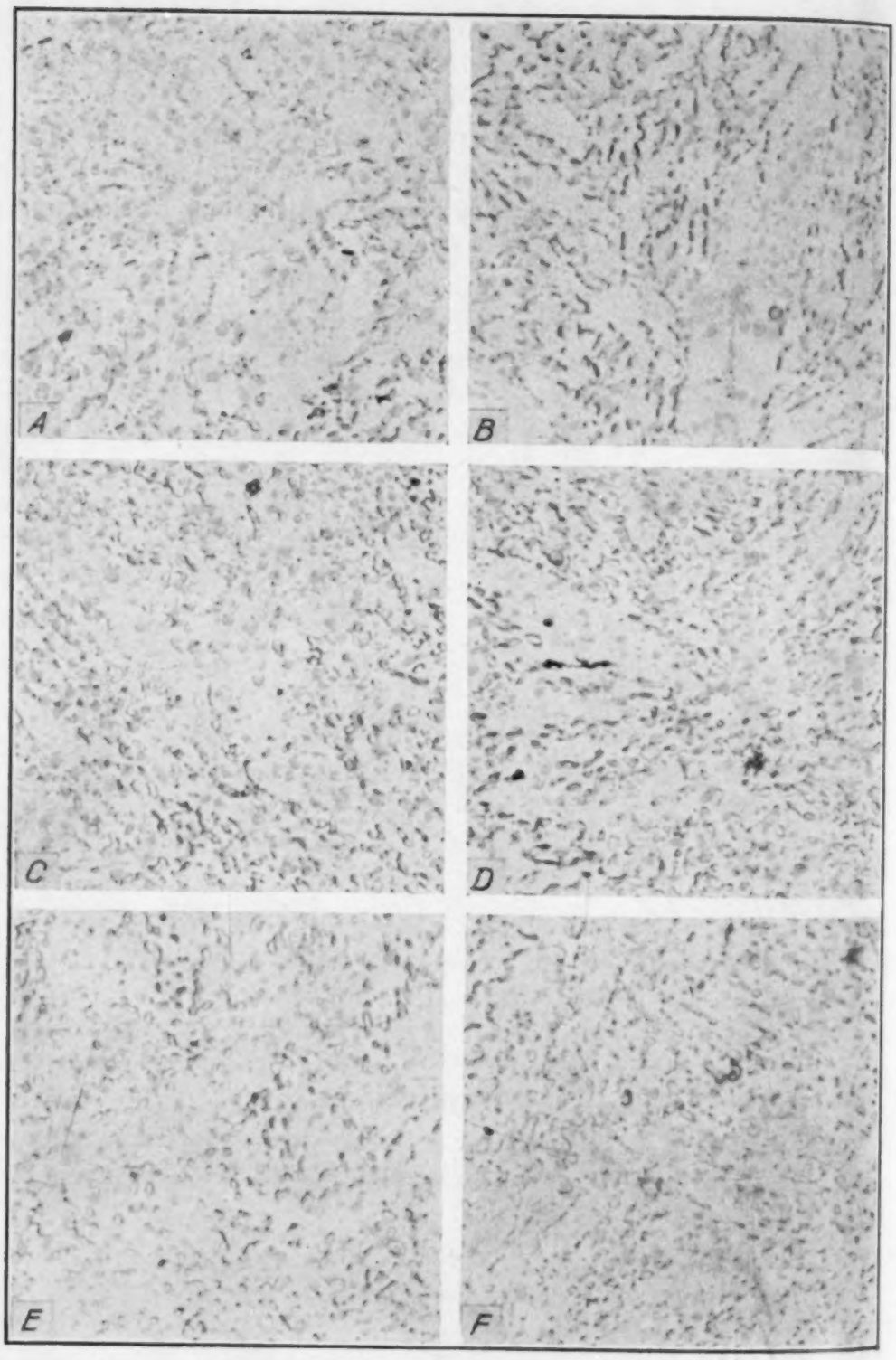


Fig. 17—Photomicrographs of Steels Tempered 125 Hours at 550 Degrees Cent. A.—Steel A. B.—Steel B. C.—Steel D. D.—Steel C. E.—Steel F. F.—Steel E. Picric Acid Etch. × 2000.

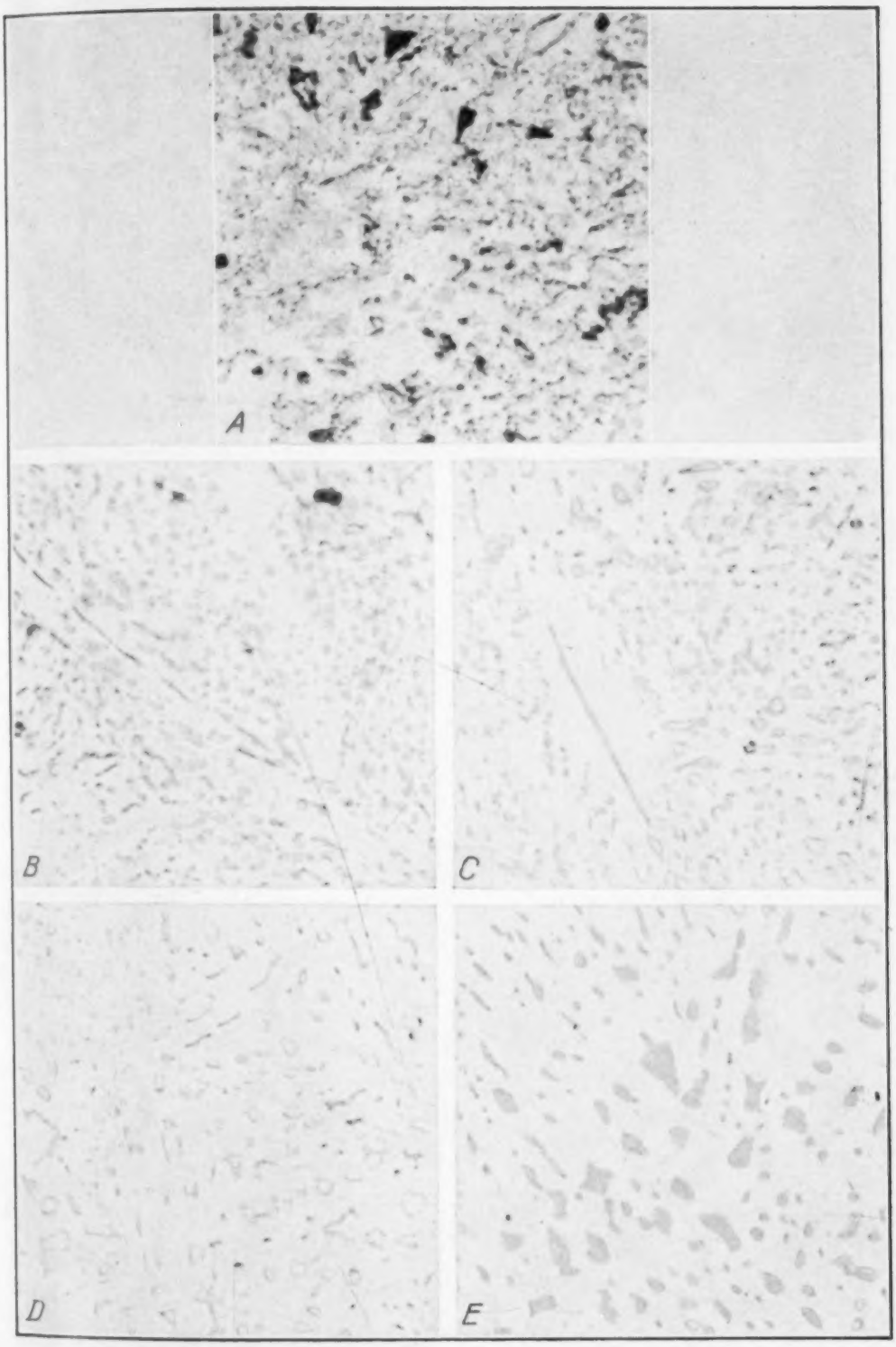


Fig. 18—Photomicrographs of Steel B Tempered at 650 Degrees Cent. A.—½ Hour. B.—5 Hours. C.—25 Hours. D.—75 Hours. E.—125 Hours. Picric Acid Etch. × 2000.

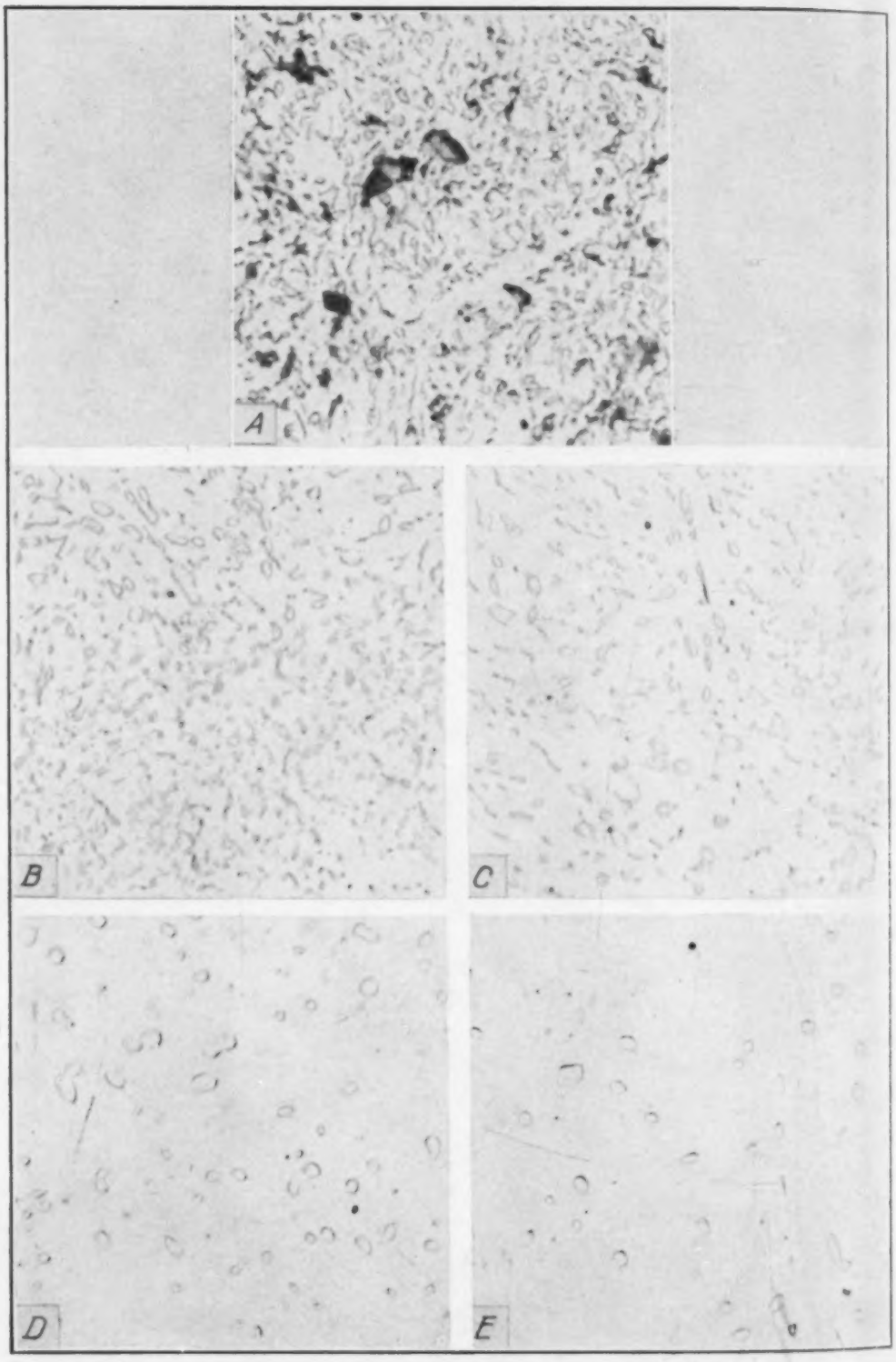


Fig. 19—Photomicrographs of Steel F Tempered at 650 Degrees Cent. A.—½ Hour. B.—5 Hours. C.—25 Hours. D.—75 Hours. E.—125 Hours. Picric Acid Etch. × 2000.

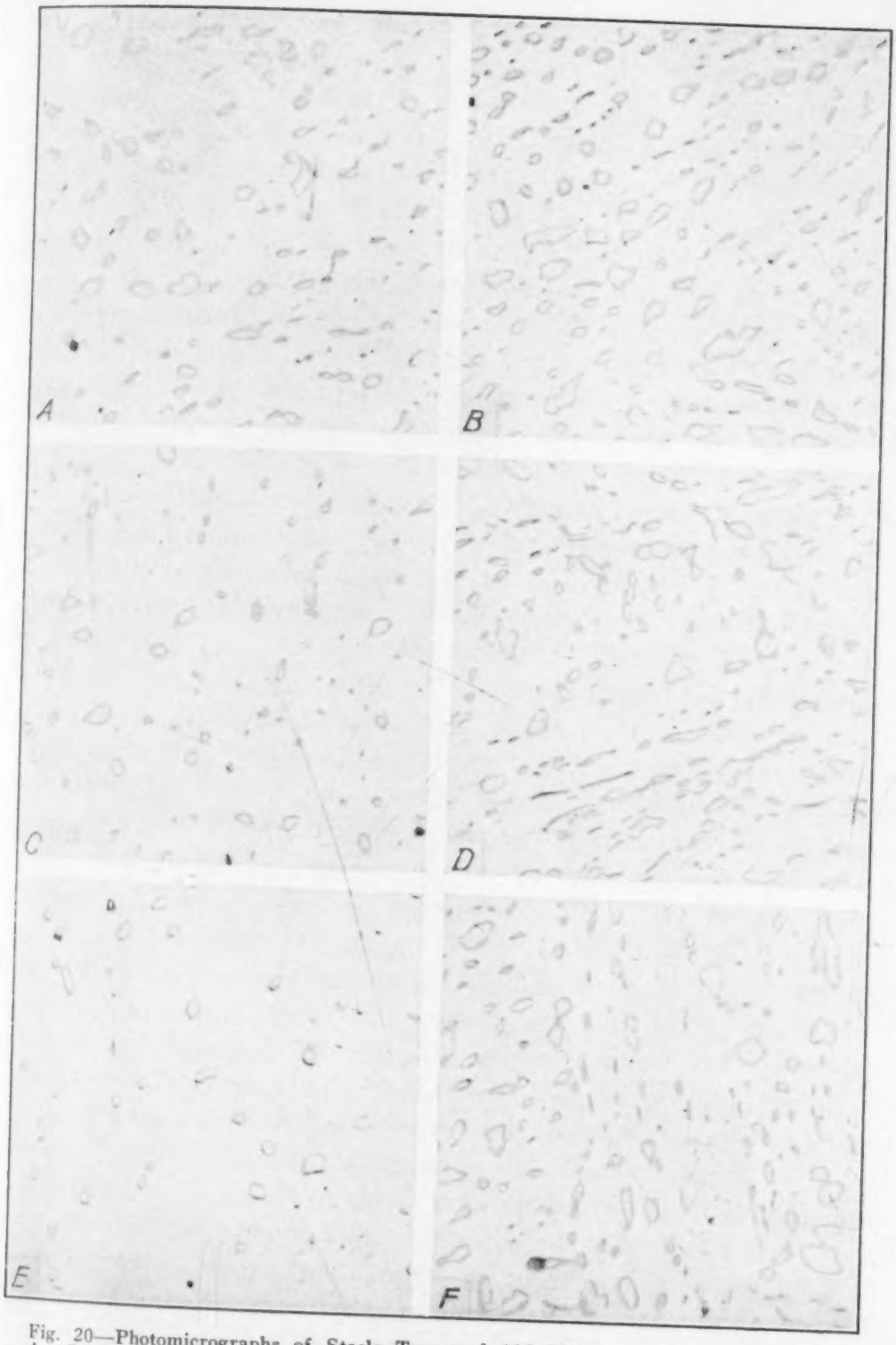


Fig. 20—Photomicrographs of Steels Tempered 125 Hours at 650 Degrees Cent. A.—Steel A. B.—Steel B. C.—Steel D. D.—Steel C. E.—Steel F. F.—Steel E. Picric Acid Etch. × 2000.

marked graphitization occurred the ferrite matrix appeared to have a dull lustre but lack of optical resolution precluded any further study. The same general characteristics are evident after tempering at 710 degrees Cent. (1310 degrees Fahr.) (Figs. 21, 22 and 23), with the exception that relatively little graphitization was noted.

In Fig. 24 unetched sections of the steels at 200 diameters are presented to illustrate the relative amounts of graphite resulting from 125 hours at 550 and 650 degrees Cent. (1020 and 1200 degrees Fahr.). Steels A, D, F show evidence of considerable dissociation of cementite at 550 degrees Cent. (1020 degrees Fahr.), and the much greater amount of graphite produced at 650 degrees Cent. (1200 degrees Fahr.) affords an explanation of the small amount of cementite observed in the etched structures at 2000 diameters (Fig. 20). Graphitization in Steels B, C, and E is considerably less evident, and the amount observed in Steel E was so small after tempering at 550 or 650 degrees Cent. (1020 or 1200 degrees Fahr.) that the unetched structures were omitted. The large graphite particles present in Steel C after 125 hours at 650 degrees Cent. (1200 degrees Fahr.) (Fig. 24J) were only present within a small area at the center of the sample. A structure similar to that of B was present outside of this area. The growth of the large graphite particles may have been hastened by some form of segregation. Very little graphitization took place in any of the steels when tempered at 710 degrees Cent. (1310 degrees Fahr.).

A comparison in the development of free graphite in Steels B and F during the time of tempering at 650 degrees Cent. (1200 degrees Fahr.) is given in Fig. 25. After ½ hour at temperature both steels exhibit the precipitation of a large amount of minute graphite particles which largely disappear in B and almost entirely disappear in F on increasing the tempering time to 5 hours. On more prolonged tempering the graphite in B continues to decrease and after 125 hours relatively little graphitization is evident. In the case of Steel F the reverse process seems to obtain so that after 125 hours the amount of carbide is remarkably small (Fig. 19) whereas the free graphite is present in large nodules and in considerable quantity (Fig. 25). Photomicrographs of Steel F after tempering 5 and 125 hours at 550 degrees Cent. (1020 degrees Fahr.) have also been included in Fig. 25 in order to show the relative amounts of graphite present at 550 and 650 degrees Cent. (1020 and 1200 degrees Fahr.).

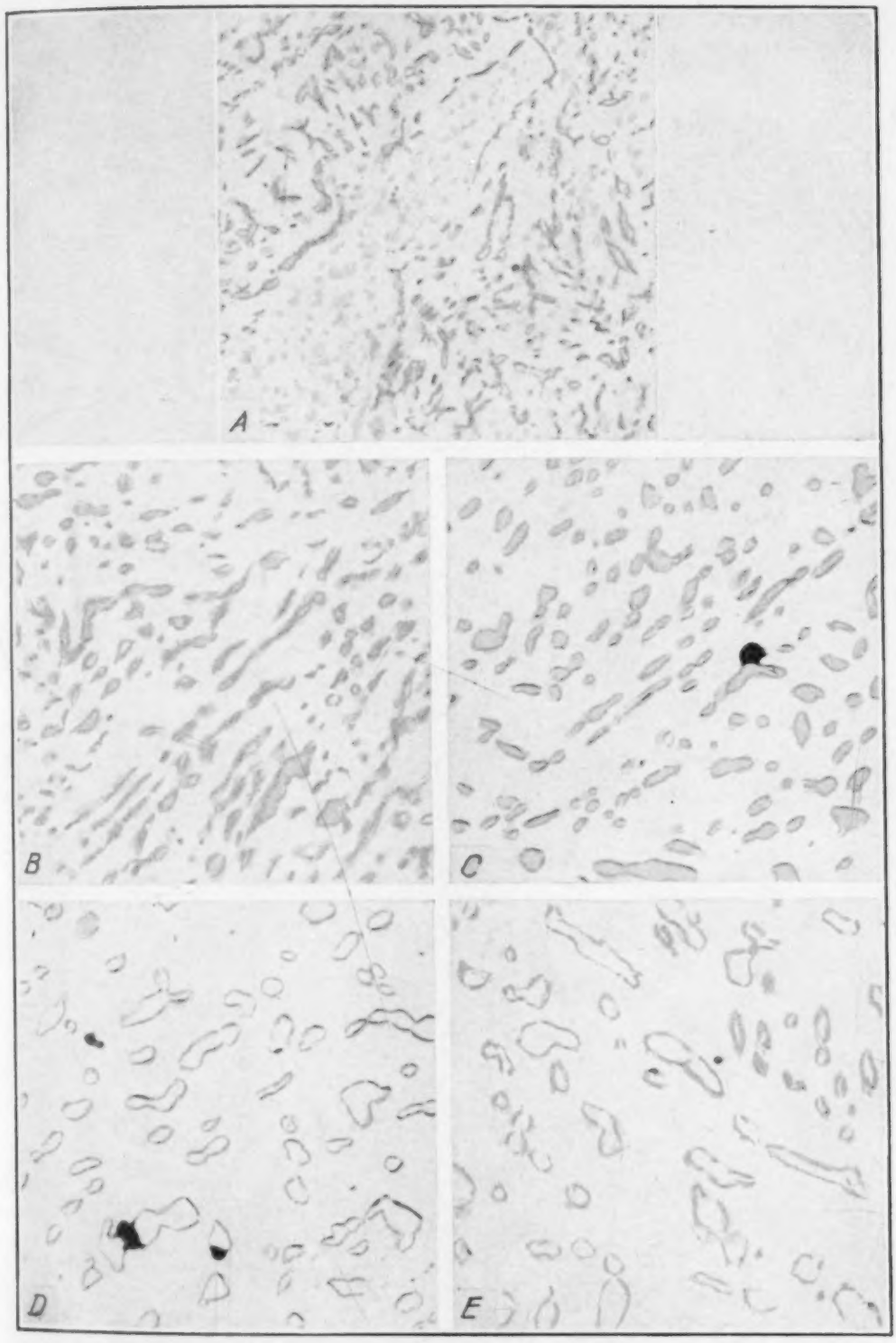


Fig. 21—Photomicrographs of Steel B Tempered at 710 Degrees Cent. A.—½ Hour. B.—5 Hours. C.—25 Hours. D.—75 Hours. E.—125 Hours. Picric Acid Etch. × 2000.

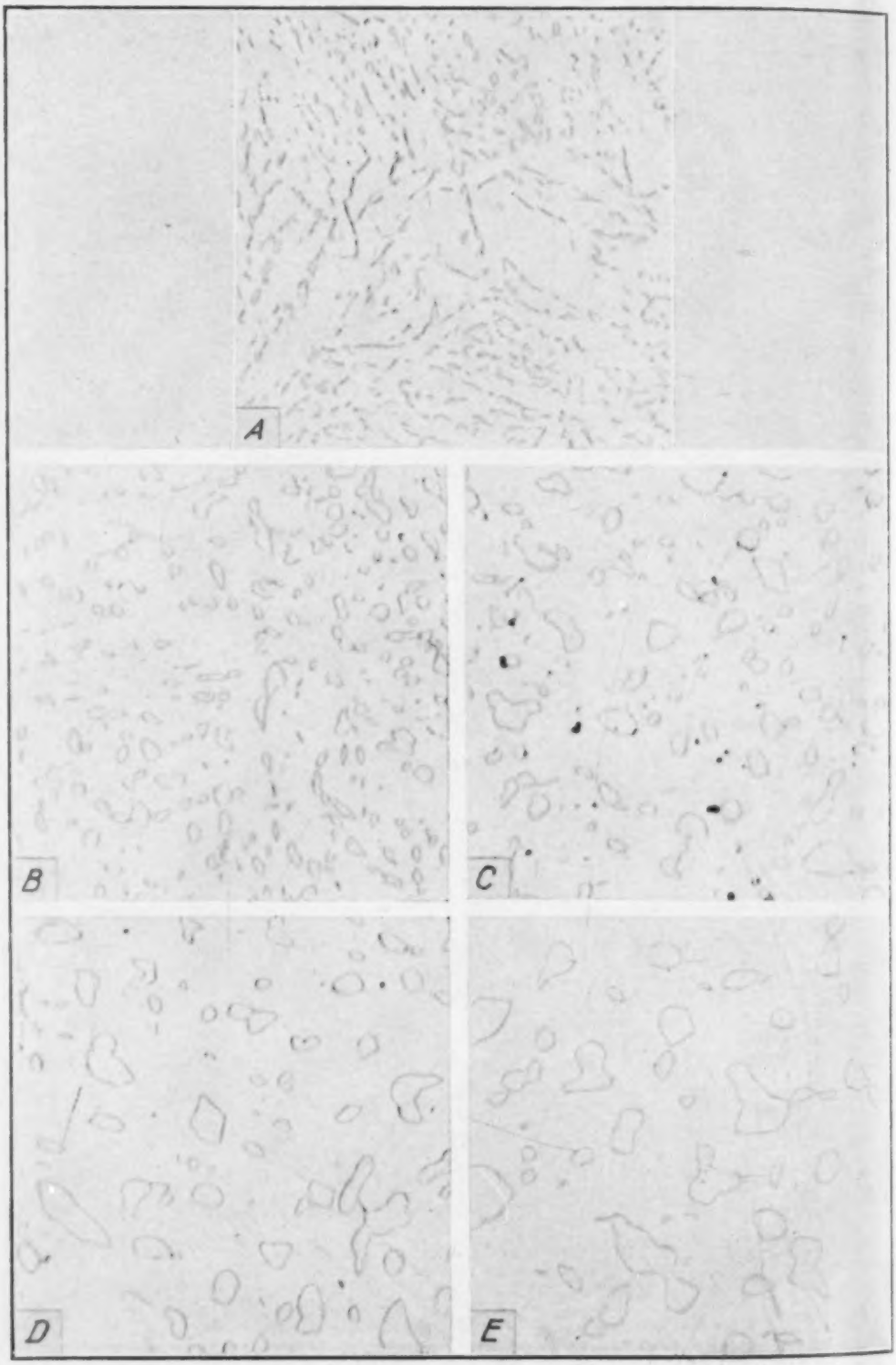


Fig. 22—Photomicrographs of Steel F Tempered at 710 Degrees Cent.
A.—½ Hour. B.—5 Hours. C.—25 Hours. D.—75 Hours. E.—125 Hours.
Picric Acid Etch. × 2000.

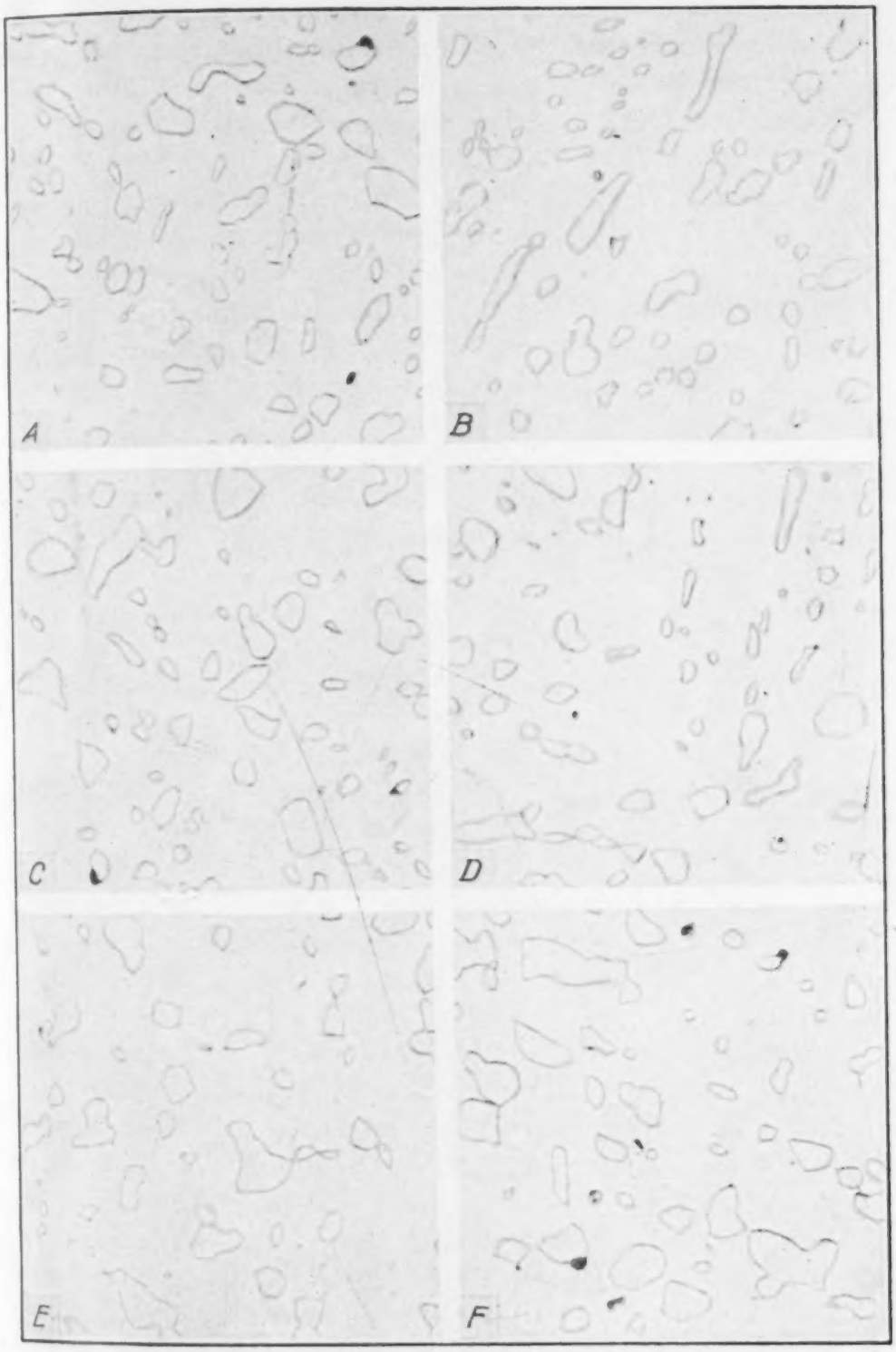


Fig. 23—Photomicrographs of Steels Tempered 125 Hours at 710 Degrees Cent. A.—Steel A. B.—Steel B. C.—Steel D. D.—Steel C. E.—Steel F. F.—Steel E. Picric Acid Etch. × 2000.

In order to confirm a presumed satisfactory correlation between the microstructures obtained from the hardness specimens, and those resulting from heat treatment of the resistivity-coercive force bars, sections were cut from the latter test rods after the final 125-hour treatment at 550, 650 and 710 degrees Cent. (1020, 1200 and 1310 degrees Fahr.). It will be recalled that the hardness specimens were subject only to one heat treatment, whereas the resistivity bars were repeatedly heated to and quenched from a constant temperature until a total tempering time of 125 hours had been completed. Typical microstructures obtained from some of the latter specimens after 125 hours at each of the three tempering temperatures are reproduced in Fig. 26.

Some interesting results are obtained when these photomicrographs are compared with those obtained from the comparable samples used in the hardness tests and tempered for similar periods (Steels B and F in Figs. 17, 20 and 23).

At 550 degrees Cent. (1020 degrees Fahr.) the structures are quite similar although the carbides appear to be slightly smaller in the resistivity specimens. A more striking difference is found in the 650 degrees Cent. (1200 degrees Fahr.) structures, where much more carbide is present in the hardness specimens than in the resistivity specimens (cf. Figs. 20B and 20E with Figs. 26D and 26E for Steels B and F). The repeated heating and cooling to which the specimens shown in Fig. 26 were subjected has apparently increased the rate of decomposition of the carbide particles. Fig. 24H, however, appears to contradict this conclusion when Steel F is considered, since it appears to have more graphite than shown in Fig. 26H.

It was observed in several of the resistivity specimens which graphitized markedly that a peripheral band developed, during the sequence of tempering operations, where much of the carbide had disappeared but little graphite could be observed. Similarly, in the core the amount was frequently insufficient to account for all the carbide which had disappeared and hence apparently dissociated. The only tentative suggestion, which can be introduced at this time, is that some volatilization of graphite occurred during the 125-hour tempering treatment.

At 710 degrees Cent. (1310 degrees Fahr.) the two samples of Steel B (Figs. 23B and 26J) are very similar. However, this is not true for F since the carbides are less equiaxed in the hardness than

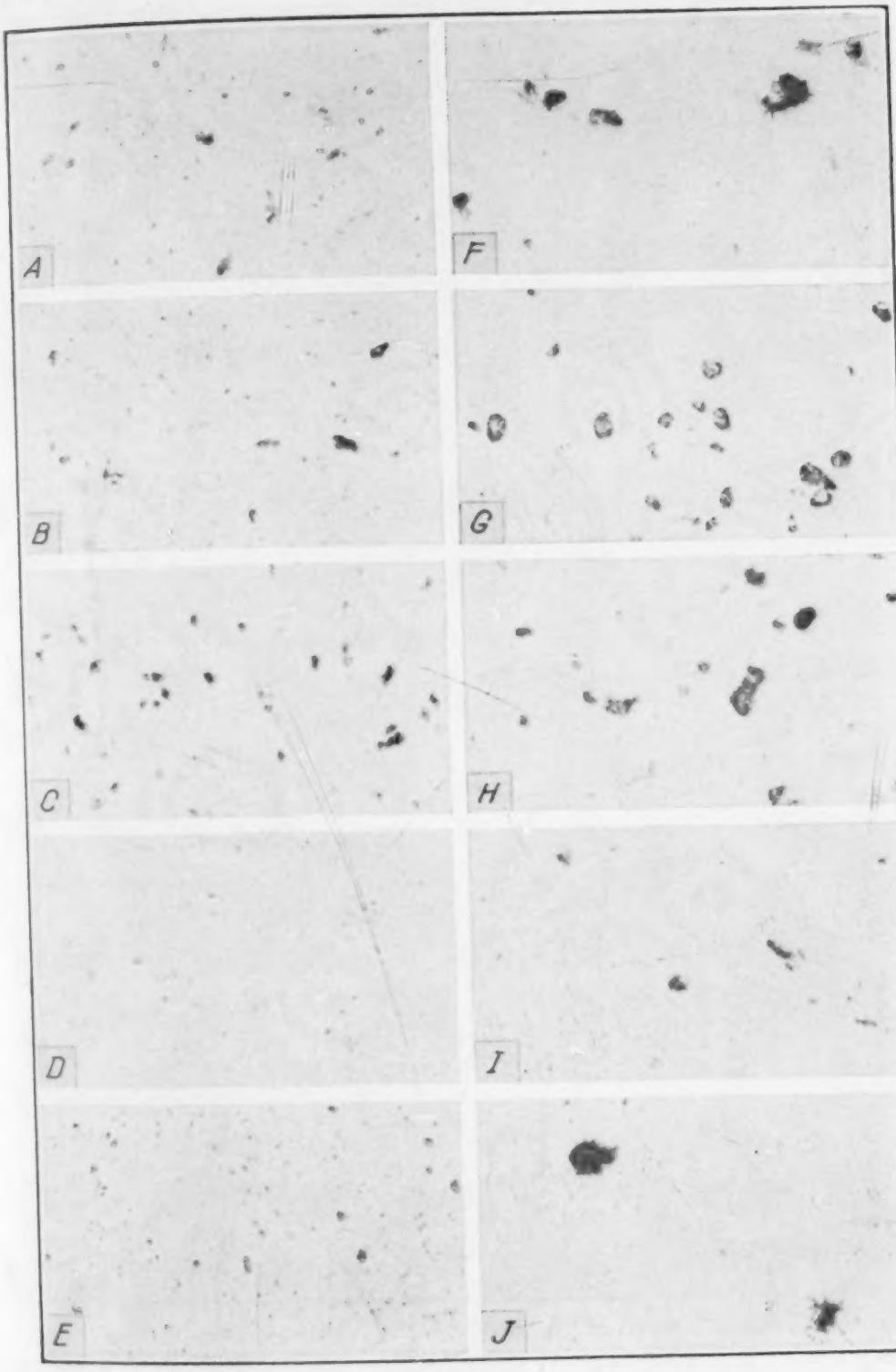


Fig. 24—Photomicrographs of Steels Tempered at 550 or 650 Degrees Cent. for 125 Hours.

At 550 Degrees Cent.: A., Steel A. B., Steel D. C., Steel F. D., Steel B. E., Steel C. At 650 Degrees Cent.: F., Steel A. G., Steel D. H., Steel F. I., Steel B. J., Steel C. Unetched. × 200.

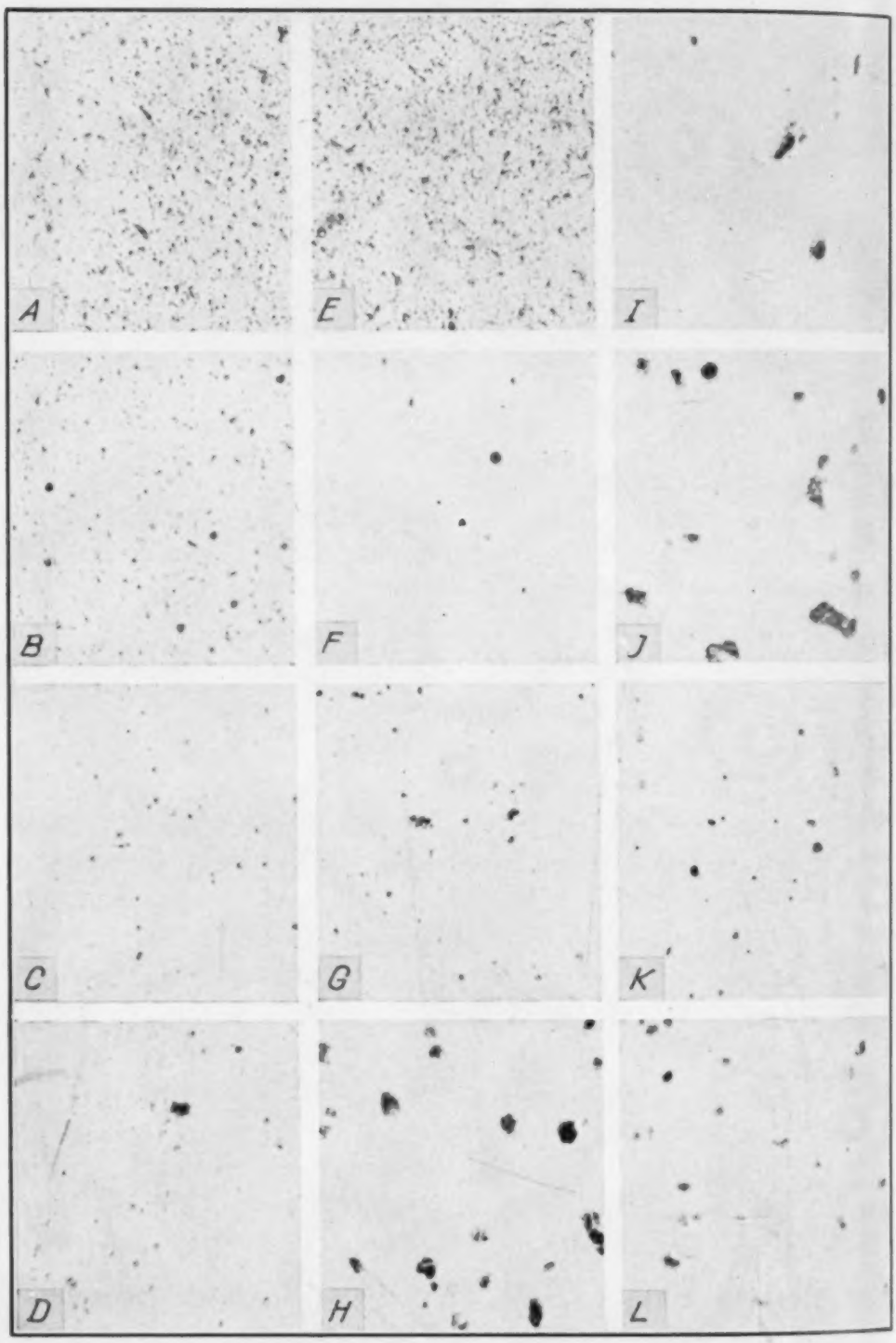


Fig. 25—Photomicrographs of Steels B and F as Tempered.

Steel B at 650 Degrees Cent.: A.—½ Hour. B.—5 Hours. C.—25 Hours. D.—75 Hours. I.—125 Hours.

Steel F at 650 Degrees Cent.: E.—½ Hour. F.—5 Hours. G.—25 Hours. H.—75 Hours. J.—125 Hours.

Steel F at 550 Degrees Cent.: K.—5 Hours. L.—125 Hours.
Unetched. × 200.

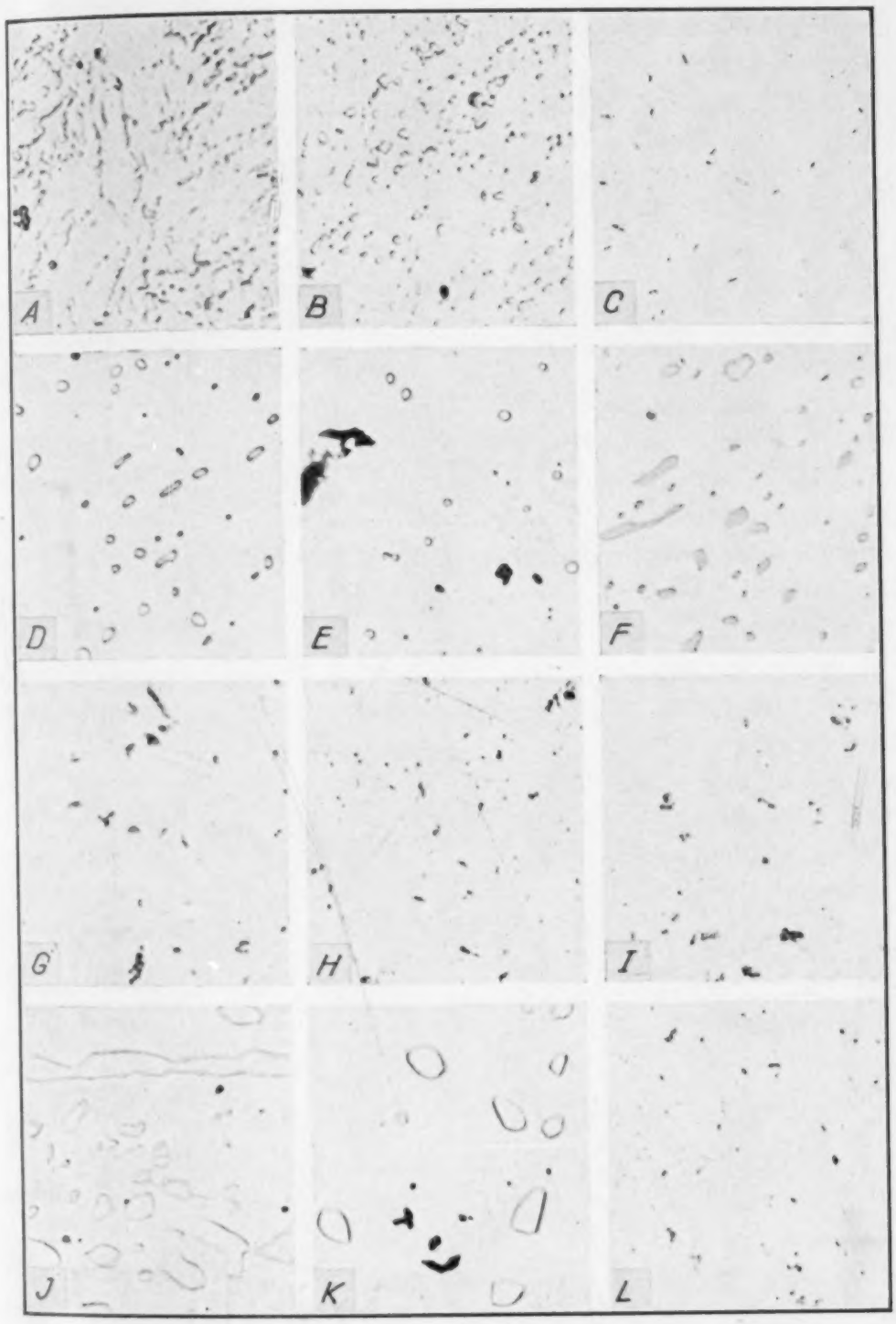


Fig. 26—Photomicrographs of Resistivity Specimens as Tempered 125 Hours.

At 550 Degrees Cent.: A.—Steel B. B.—Steel F. Etched. × 2000.
C.—Steel F. Unetched. × 200.

At 650 Degrees Cent.: D.—Steel B. E.—Steel F. F.—Steel E. Etched. × 2000.
G.—Steel B. H.—Steel F. I.—Steel E. Unetched. × 200.

At 710 Degrees Cent.: J.—Steel B. K.—Steel F. Etched. × 2000.
L.—Steel F. Unetched. × 200.

in the resistivity sample (Figs. 23E and 26K). This difference is reflected in the final Rockwell values for the steels—hardness sample 88B and resistivity sample 82B.

Study on Austenitic Grain Size

In view of the important rôle which austenitic grain size has been shown to have in consideration of reaction rates, it is of interest to discuss the results of a study which the authors gave to the austenitic grain size of the steels examined in the present investigation. As indicated in a previous section it was impossible to correlate the grain size, supplied by the companies, of all the steels since some of the values were determined by the A.S.T.M. and others by the Shepherd test. Since the authors consider the Shepherd method of determining grain size a more rational procedure this latter test was utilized. Quite recently Herty has discussed this problem and the authors are in accord with his views.

Since Shepherd (8), Digges and Jordan (30) and others have observed that the condition of the carbides prior to hardening may have a decided influence upon apparent austenitic grain size, it was decided to include this problem in our study of the six steels discussed. In this work we were fortunate to have the kind cooperation of Mr. G. V. Luerssen of the Carpenter Steel Company who supervised the determination of the grain size values.

The six steels were first given the following preheating treatment:

> Treatment X: 30 minutes at 870 degrees Cent. (1600 degrees Fahr.), oil-quenched, annealed 1 hour at 710 degrees Cent. (1310 degrees Fahr.), cooled 5 degrees Cent. (9 degrees Fahr.) per hour down to 650 degrees Cent. (1200 degrees Fahr.) and air-cooled.
> Treatment Y: 5 minutes at 930 degrees Cent. (1700 degrees Fahr.) and oil-quenched.

Treatment X was used to promote large carbide particles while treatment Y was employed to retain small particles.

Specimens 3¼ inch long and ⅜ inch in diameter (bar stock) were subjected to treatment X and then machined to $\frac{11}{32}$ inch round to remove any decarburized layer. Specimens for treatment Y were machined to a $\frac{11}{32}$-inch diameter prior to heat treatment. All specimens were cut to a 2-inch length before hardening.

After pretreatment X or Y, one specimen of each steel was hardened by the authors and by the Carpenter Steel Company in

Fig. 27—Appearance of the Fractures of the Specimens Used for Fracture-Grain Size Determination.

order to permit comparison between the results obtained by the two laboratories. Three methods of hardening were employed.

1. Annealed 5 minutes at 790 degrees Cent. (1450 degrees Fahr.) followed by quenching in 10 per cent brine solution.
2. Annealed 5 minutes at 845 degrees Cent. (1550 degrees Fahr.) followed by quenching in 10 per cent brine solution.
3. Annealed 60 minutes at 1000 degrees Cent. (1830 degrees Fahr.) followed by a quench in 10 per cent brine.

Mr. Luerssen supervised all determinations for fracture grain size. The data are listed in Table VI where the larger numbers correspond to small grain size. The small diameter of specimen precluded any measurements being made for standard hardness penetration.

A comparison of the appearance of the fractures is shown in Fig. 27. Experience indicates that the fracture numbers can be read within an approximate accuracy of $\pm$ ¼ of a number. This fact suggests that data differing by not more than ½ number may be regarded as indicating a similar grain size within experimental error.

A comparison of the authors' values with those resulting from heat treatment by the Carpenter Steel Company, shows the latter values to be slightly higher. With the exception of Steel D, when given treatment X and hardened at 790 degrees Cent. (1450 degrees Fahr.), all values may be considered comparable, within the limits of reproducibility. Eighty per cent of the data checked to within ¾ of a number.

The variation in prehardening treatments appears to have had little effect upon the grain size, but the effect of change in hardening temperature is at once evident. In Table VII the mean of the values obtained from the heat treatments in the two laboratories has been recorded and, in order to permit ready comparison of the reaction of the steels to grain growth, a numeral indicating order of coarseness has been added. Thus the steel with the finest grain size for each of the six heat treatments has been designated "1" while the one with coarsest grain in each of these treatments has been designated "6." Obviously numbers 3 and 4 indicate a mean grain size. However, these numbers indicated only the order of grain size from coarsest (No. 6) to finest (No. 1) and not the magnitude of the difference, for each of the six heat treatments.

Using this notation in Table VII one may observe the interesting fact that the steels have practically the same order (fine to coarse grain), namely F, C, A, D, B and E, irrespective of the

Table VI
Fracture Grain Size

Hardening[3] Temperature		Hardened by Pennsylvania State College Steel Designation						Hardened by Carpenter Steel Co.				
Degrees Fahr.	Degrees Cent.	A	B	C	D	E	F	A	B	D	E	F
					Treatment X[1]							
1450	790	8¾	8	9¼	8¼	7	9¼	8¼	7½	7	7¼	9¼
1550	845	6¾	6	8¼	6	5	9	6	5½	6¼	5	9¼
1830	1000	3	3	4½	3¾	2½	5½	2¾	2½	3¼	2¾	4¾
					Treatment Y[2]							
1450	790	9¼	8¼	9¼	8	6	9¼	8½	7¾	7	7	9
1550	845	7	6½	8½	6½	5	9	6	6	6¼	4¾	8
1830	1000	3	2¾	4	3½	3	4¾	3	2¼	3	2¾	4

1—Prehardening treatment X:—
30 minutes at 1600 degrees Fahr. (870 degrees Cent.) oil quenched, annealed 1 hour at 1310 degrees Fahr. (710 degrees Cent.), cooled 9 degrees Fahr. (5 degrees Cent.) per hour to 1200 degrees Fahr. (650 degrees Cent.), air cooled.

2—Prehardening treatment Y—:
5 minute at 1700 degrees Fahr. (930 degrees Cent.), oil quenched.

3—Hardening treatment after treatment X or Y:—
1450 degrees Fahr. (790 degrees Cent.) or 1550 degrees Fahr. (845 degrees Cent.), 5 minutes at temperature, quenched in 10 per cent brine.
1830 degrees Fahr. (1000 degrees Cent.), 60 minutes at temperature, quenched in 10 per cent brine.

heat treatment used. Steel F, when subjected to any of the treatments used in this investigation, has the finest austenitic grain size of the series, while steel E usually has the coarsest grain.

The microstructures of two of the steels have been included to illustrate typical structures observed after treatment X and after subsequently quenching from 1000 degrees Cent. (1830 degrees Fahr.) after 1 hour at temperature in 10 per cent brine solution (Fig. 28). Figs. 28A and 28B typify the structures present in steels B and F after prehardening treatment X. Although, on the whole, both steels had carbides of the same size, steel B was more prone to form massive particles. Some of the massive particles are shown in Fig. 28A.

Fig. 28C, showing the quenched structure of B, illustrates the very large martensitic needles which were common in this steel. The needles of steel F (Fig. 28D) were always much smaller. This is what might be expected since steel B had coarse austenite grains while F had small grains.

General Comments

In this investigation it has been shown how the reaction to tempering of quenched hypereutectoid steels may differ profoundly in

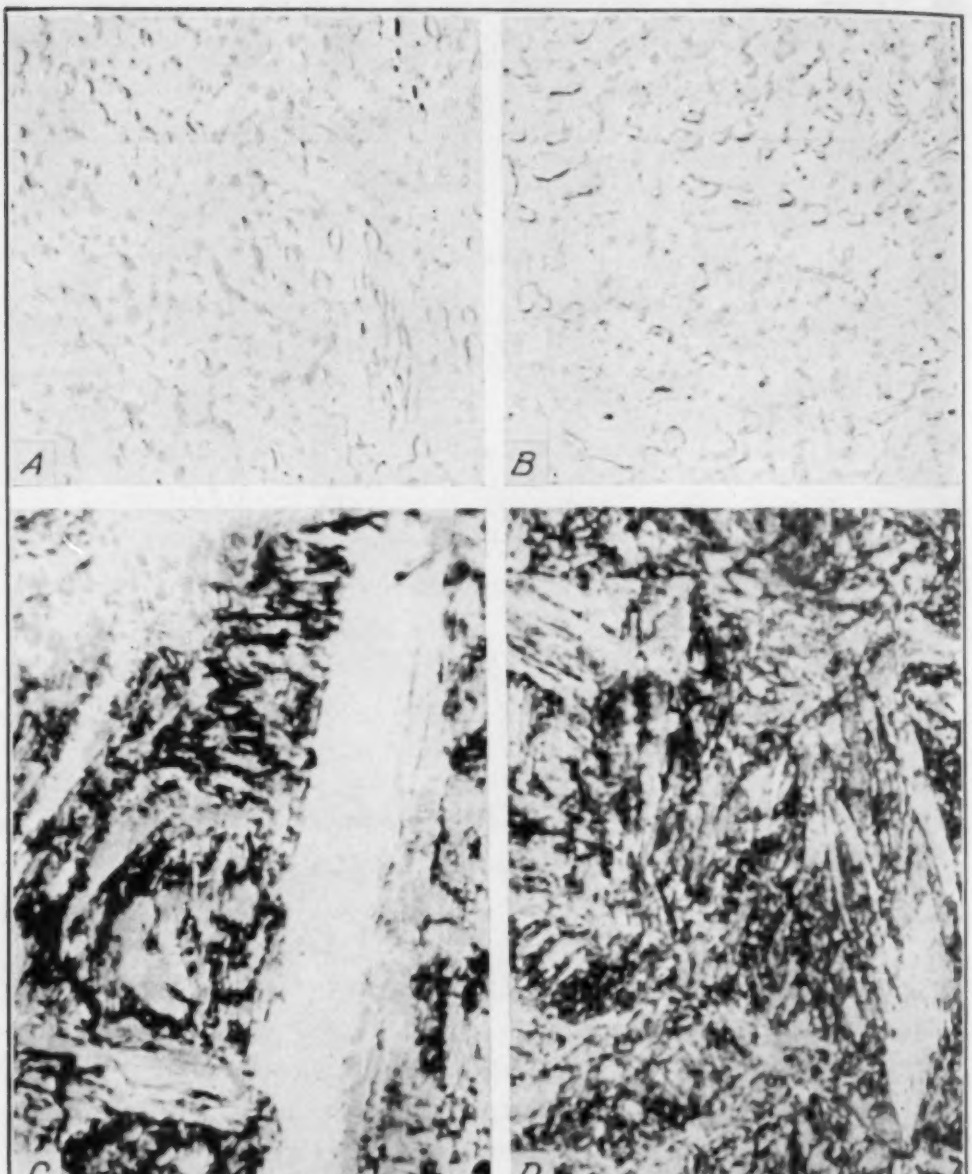


Fig. 28—Photomicrographs from Specimens Used for Fracture-Grain Size Determinations.
A.—Steel B. B.—Steel F. After Prehardening Treatment X.
C.—Steel B. D.—Steel F. After Prehardening Treatment X.
and Subsequent Brine Quench After 1 Hour at 1000 Degrees Cent.
Picric Acid Etch. × 2000.

six steels of "similar" chemical analysis. Differences in reaction rate and in mode of reaction have been shown to be reflected in the progressive change, with time at temperature, of electrical resistivity, coercive force and hardness measurements. Some of the reasons for the variations observed have been clearly revealed during the studies on the microstructural characteristics of the steels.

Table VII
Analysis of Fracture Grain Size Data

Hardening Temperature Degrees Fahr.	Degrees Cent.	Average Values[1] Steel Designation A	B	C	D	E	F	Order of Coarseness[2] Steel Designation A	B	C	D	E	F
					Treatment X								
1450	790	8½	7¾	9¼	7¾	7¼	9¼	3	4	1	4	6	1
1550	845	6½	5¾	8¼	6¾	5	9¼	3	5	2	4	6	1
1830	1000	3	2¾	4½	3½	2¾	5	4	5	2	3	5	1
					Treatment Y								
1450	790	8¾	8	9¼	7½	6½	9¼	3	4	1	5	6	1
1550	845	6½	6¼	8½	6½	5	8½	3	5	1	3	6	1
1830	1000	3	2½	4	3¼	3	4½	4	6	2	3	4	1

1—Average of fracture grain sizes—, Table VI, obtained by The Pennslyvania State College and Carpenter Steel Company.

2—"1" indicates the steel with the highest fracture grain size value (finest grain) for each of the given temperatures. "6" designates the steel having the lowest value (coarsest grain) for each of the temperatures.

Note:—No significance is attached to these numbers when any two different hardening temperatures or treatments are compared.

While the factors which control and influence the changes in magnitude of the physical properties studied are not well understood, present knowledge appears to indicate that the most important factors controlling these properties are as follows:

Coercive force. Metallic materials having a high coercive force are heterogeneous substances containing some phase or phases in a high degree of dispersion. Hence, small dispersed particles produce a high coercive force. When quenched carbon steels are tempered, the coercive force progressively decreases as the time or temperature of annealing increases. In other words, the coercive force decreases as the size of the carbides increases and their total surface area decreases. Thus, it may be assumed that measurements of this physical property should afford a ready means of following the spheroidization of iron carbide during the tempering of quenched martensitic carbon steels. It should be a quantitative method which would be particularly valuable when the aggregation of carbide particles is too small for resolution under the microscope.

Electrical resistivity. This property is largely controlled by the amount of the elements present in solid solution in the metallic conductor. The state of aggregation of a dispersed phase, following its precipitation, probably has little influence upon the resistivity. However, since it is well known that solubility depends upon particle size, a decrease in solubility, accompanying spheroidization in

carbon steels, may be expected to be revealed by a progressive decrease in electrical resistivity. However, this decrease is likely to have a relatively small magnitude.

Hardness. This property has long been used to follow the progress of spheroidization. The structural change which influences hardness is essentially dependent on diffusion and the relationship should be expressed by an exponential function such as proposed by Bailey and Roberts (2). Any deviation from the simple linearity between hardness and log time of tempering indicates the introduction of some new factor into the diffusion mechanism.

Quenched steels which have previously had coarse austenitic grains have a lower coercive force than those which have had fine grains. This is probably due to the fact that more complete precipitation occurs in the fine-grained steels during quenching. The condition no longer holds as soon as the quenched structure is tempered. Since coercive force of tempered steels is primarily dependent on the quantity and size of the carbides, it is greatly reduced when graphitization takes place.

The electrical resistivity of quenched or tempered steels is largely influenced by the silicon content. Those which have the greater quantity of silicon also have the higher resistivity.

The hardness of tempered steels appears to be influenced by factors which are similar to those that affect coercive force.

The change in coercive force is linear with respect to the logarithm of the tempering time and its rate of change increases with increase in temperature. These relationships are only true so long as the carbides do not decompose.

From the limited amount of data available for comparison, some evidence was obtained which indicated that there might be a linear relationship between coercive force and carbide surface area such as Gottschalk (23) had predicted. However, it is felt that this evidence only gave an indication of the linearity and further work should be done in order to verify this relationship.

The change in resistivity with time is an exponential function, and the rate of the change is dependent on temperature. This rate is markedly greater at 550 and 650 degrees Cent. (1020 and 1200 degrees Fahr.), where carbon solubility in alpha iron is low (36), than it is at 710 degrees Cent. (1310 degrees Fahr.). In a summary of the factors which influence diffusion, Mehl (35) has stated that a solute will diffuse more rapidly through a solution that has a

Table VIII

Hardness of Resistivity-Coercive Force Specimens After 125 Hours at the Given Temperatures

Rockwell B

	550 Degrees Cent.	Average	650 Degrees Cent.	Average	710 Degrees Cent.	Average
A	94.0	....	73.2	....	85.6	....
	92.3	93.1	73.2	73.2	86.0	85.8
B	100.4	....	79.4	....	86.2	....
	101.0	100.7	75.3	77.3	89.4	87.8
C	97.0	....	75.4	....	83.8	....
	98.1	97.5	73.5	74.4	82.6	83.2
D	95.7	....	71.7	....	83.0	....
	95.4	95.5	....	71.7	89.0	86.0
E	97.0	....	79.3	....	88.9	....
	95.8	96.9	78.8	79.0	86.4	87.6
F	93.3	....	73.5	....	81.7	....
	93.1	93.2	78.4	75.8	89.6	85.6

low solubility for the solute, than it will through one of high solubility. This fact may in part account for the greater slope of the resistivity-log time curves at 550 and 650 degrees Cent. (1020 and 1200 degrees Fahr.).

The hardness of the resistivity specimens was generally lower than that for the regular hardness samples. This is shown when the values found in Table VIII are compared with the 125 hour values previously given in Table V. The difference varied with the tempering temperature. It was fairly small at the temperatures where a limited amount of graphitization occurred, such as 550 and 710 degrees Cent. (1020 and 1310 degrees Fahr.), although the steels with the greater tendency to graphitize had the slightly larger difference in hardness. When they were tempered at 650 degrees Cent. (1200 degrees Fahr.), where the amount of graphitization was large, the difference in the two sets of hardness readings was considerable for the steels less prone to graphite formation, and small for those that were easily graphitized. This is undoubtedly due to the fact that the repeated heating and cooling, to which the resistivity specimens were subjected, increase the rate of dissociation of iron carbide to the greatest extent in the steels which were usually quite resistant to this phenomenon.

The rate at which carbides spheroidize depends upon their condition in the quenched structure. Those which precipitate in structures characterized by large martensitic needles are slower in assuming an equiaxed condition than those which form from small needle structures. This is probably due to the tendency of the carbides to precipitate in large quantities on certain crystallographic planes in

the coarse martensitic needle structure since large carbide masses are more difficult to spheroidize than small ones. The rate of spheroidization is also dependent upon the factors that control graphitization since the carbides change their shape for one of an equiaxed condition more rapidly in the steels that are easily graphitized than in those which are resistant to this phenomenon.

Carbides may grow to large sizes without acquiring an equiaxed shape. This is most prevalent in steels that have high resistance to graphitization.

Graphitization takes place more readily near 650 degrees Cent. (1200 degrees Fahr.) than it does at temperatures which are just below the lower critical point. Repeated heating and cooling from temperatures below the critical range also increase the rate at which this reaction occurs. The greater rate of graphitization in some steels may be accounted for by the presence of large quantities of metallic aluminum. Thus, steels A and F, which have the highest metallic aluminum contents, exhibit a strong tendency to graphitize. It is difficult to evaluate from the present work what effect the high temperature quench may have on this phenomenon, although previous investigators have observed that increase in quenching temperature increases the rate of graphitization in white cast irons.

Much evidence has been presented in this investigation to show that steels of similar analysis may react quite differently during tempering. Even though it is fully realized that all factors which promote dissimilarity of reaction, have not been isolated, it is considered that some should be discussed. Probably the most important factors are those which promote dissociation of the iron carbide. These factors tend to speed up spheroidization as well as the decomposition of the carbide particles. The presence of metallic aluminum in the steel appears to have a marked affect in promoting or accelerating these phenomena.

Austenitic grain size also has been observed to have an influence on the behavior of the steels during tempering. This factor appears to exert its influence by controlling the nature of the quenched structure which in turn regulates the manner in which the carbides precipitate and further react during tempering.

When the "fracture grain size specimens" were examined under the microscope it was noticed that the size of the martensitic needles varied with the size of the austenitic grains from which they were formed. Large needles were formed from large austenite

grains, and small ones were produced from small grains. However, steel E was an outstanding exception to this observation since its fracture grain size value indicated that it was a coarse-grained steel, while its quenched structure, exhibiting small martensite needles, resembled that of a fine-grained steel. Since the fracture indicated coarse grain, a low aluminum oxide content might be anticipated in the chemical analysis. An examination of Table I shows that such is not the case, as the steel has the second highest alumina content recorded for the series of six steels. In other words, the behavior of this steel on quenching (with respect to microstructure) and on subsequent tempering, followed the reaction indicated by its alumina content more closely than did the fracture grain size (Shepherd test).

It is hoped that additional data will be available at an early date to illustrate further the dissimilarity in reaction of steels to tempering.

Bibliography

1. H. C. Ishen, "The Spheroidization of Cementite," *Heat Treating and Forging,* July 1922, p. 300-303.
2. R. W. Bailey and A. M. Roberts, "Testing of Materials for Service in High-Temperature Steam Plant," *Institute of Mechanical Engineers,* Vol. 122, 1932, p. 209-284.
3. J. A. Duma, "Spheroidizing Steels," *Heat Treating and Forging,* Dec. 1934, p. 583-5.
4. J. H. Whiteley, "The Formation of Globular Pearlite," *Journal,* Iron and Steel Institute, No. I, 1922, p. 339-357.
5. J. H. Kelley, "A Study of Spheroidization of Hypereutectoid Steels and Thermal Gradients," Thesis, The Pennsylvania State College, 1935.
6. E. C. Bain, "Factors Affecting the Inherent Hardenability of Steel," TRANSACTIONS, American Society for Steel Treating, Nov. 1932, p. 385-428.
7. E. S. Davenport and E. C. Bain, "General Relations Between Grain Size and Hardenability and the Normality of Steels," TRANSACTIONS, American Society for Metals, Dec. 1934, p. 879-925.
8. B. F. Shepherd, "The P-F Characteristics of Steel," TRANSACTIONS, American Society for Metals, Dec. 1934, p. 979-1016.
9. C. H. Herty and B. M. Larsen, "Abnormality in Case-Carburized Steels," Cooperative Bulletin No. 45, Carnegie Institute of Technology, U. S. Bureau of Mines, Mining and Metallurgical Advisory Boards.
10. C. H. Herty, D. L. McBride, and E. H. Hollenback, "Which Grain Size," TRANSACTIONS, American Society for Metals, March 1937, p. 297-314.
11. H. W. McQuaid, "The Importance of Aluminum Additions in Modern Commercial Steels," TRANSACTIONS, American Society for Metals, Dec. 1935, p. 797-838.
12. F. Rapatz and W. Wrazej, "Steels Unsusceptible and Susceptible to Heat Treatment," Hutnik, 1937, Vol. 9, No. 1, p. 5-12. Abstracted in *Bulletin,* Iron and Steel Institute, April 1937, p. 273A-275A.
13. G. R. Brophy and E. R. Parker, "Influence of Aluminum on the Normality of Steel," TRANSACTIONS, American Society for Metals, March 1937, p. 315-324.

14. Howard A. Smith, "Reactions in the Solid State—I—Initial Course of Subcritical Isothermal Diffusion Reactions in Austenite in an Alloy Steel," American Institute of Mechanical Engineers, Iron and Steel Division, Vol. 116, 1935, p. 342-362.
15. Cleaves and Thompson, "Electric Properties of Pure Iron," The Metal-Iron Monograph, Chapter VI, VII.
16. A. S. Norbury, "The Effect of Various Elements on the Electrical Resistance of Iron," *Journal*, Iron and Steel Institute, Vol. 101, 1920, p. 627-644.
17. "Electric and Magnetic Properties of Commercial Iron-Carbon Alloys," Alloys of Iron and Carbon, Vol. II, Chapter XVI.
18. W. Hume-Rothery, "The Electrical Conductivity of Pure Metals and the Factors Which Affect It," The Metallic State, Chapter I.
19. F. C. Thompson, "Electrical and Magnetic Properties of Pure Iron with Relation to Grain Size," *Philosophical Magazine*, 1916, p. 357.
20. E. D. Campbell, "The Influence of Heat Treatment on the Specific Resistance and Chemical Composition of Carbon Steels," *Journal*, Iron and Steel Institute, No. II, 1915, p. 164-180.
21. J. A. Mathews, "Austenite and Austenitic Steels," American Institute of Mechanical Engineers, 1925, p. 568-596.
22. V. H. Gottschalk and F. S. Wartman, "Magnetization Curves for Magnetic Powders," U. S. Bureau of Mines, Report of Investigations No. 3268, Feb. 1935, p. 67-82.
23. V. H. Gottschalk, "Coercive Force of Magnetic Powders," U. S. Bureau of Mines, Report of Investigations No. 3268, p. 83.
24. C. W. Davis and Max Hartenheim, "A Simple Method for the Determination of Magnetization-Coercive Force," The Review of Scientific Instruments, March 1936.
25. A. W. F. Green, "Black Fractures in Carbon Tool Steels," *Chemical and Metallurgical Engineering*, Vol. 27, 1922, p. 265-267.
26. R. S. Archer, "Graphitization of White Cast Irons," Discussion by Emmons, *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 67, 1922, p. 445-465.
27. A. Portevin and P. Chevenard, "La Graphitisation des Acier à Basse Température," C. R. Academy Science, Paris, Vol. 193, 1931, p. 169-171.
28. A. B. Kinzel and R. W. Moore, "Graphite in Low-Carbon Steel," Metals Technology, (No. 6), Sept. 1934, (American Institute of Mining and Metallurgical Engineers, Technical Publication 565).
29. S. Epstein, "The Iron-Graphite Diagram," Chapter V, Alloys of Iron and Carbon, Vol. 1, p. 143-145.
30. T. G. Digges and Louis Jordan, "Hardening Characteristics and Other Properties of Commercial 1 Per Cent Carbon Tool Steel," *Journal of Research*, Bureau of Standards, Research Paper 837, Oct. 1935.
31. H. A. Schwartz, "Malleable Iron—Recent Progress," *Metals and Alloys*, Vol. 2, 1931, p. 143-149.
32. F. R. Bonte and Martin Fleischann, "Developments in Graphitic Steel for Tools and Dies," METAL PROGRESS, April 1937, p. 409-413.
33. "Tentative Definitions of Terms, with Units and Symbols, Relating to Magnetic Testing," American Society for Testing Materials, Tentative Standards, Part I, 1936, p. 518-523.
34. F. F. Lucas, "Further Observations on the Microstructure of Martensite," TRANSACTIONS, American Society for Metals, Vol. 15, p. 339-367.
35. R. F. Mehl, "Diffusion in Solid Metals," *Transactions*, American Institute of Mining and Metallurgical Engineers, Institute of Metals, Division of Science Lecture, Vol. 118, 1936, p. 11-56.
36. J. H. Whiteley, "A Survey of the Iron-Carbon Diagram Near Zero Carbon (Below 1000 Degrees Cent.)," *Journal*, Iron and Steel Institute, No. I, 1936, p. 377-397.

DISCUSSION

Written Discussion: By Robert S. Rose, Vanadium-Alloys Steel Company, Boston, Mass.

This paper of Messrs. Austin and Norris points out some interesting phenomena relating to the dissimilarity in their tempering reactivity of several quenched carbon steels of nearly identical chemistry. Particularly significant is the evidence that graphitization occurs most rapidly at about 1280 degrees Fahr. resulting in depletion of carbides which seems to satisfactorily account for the subnormal hardness exhibited by Steels A — D — F and X when tempered in this range. However, since Steels B — C — E show photomicrographic evidence of some graphitization, we wonder how the authors reconcile this with the linear decrease of hardness with increasing temperature as shown by these steels. Is it, perhaps, that the loss of hardness resulting from a small amount of graphitization is more than compensated by the greater loss of hardness due to carbide coalescence at slightly higher tempering temperatures?

The authors' inferential conclusion that Fig. 3 (depicting the hardness distribution of Steel X) refutes the assumption "that the decrease in hardness of a quenched high carbon steel increases with the tempering temperature until the critical range is reached" is controvertible and not justified by the evidence they present. First of all it must be conceded that this assumption is based upon practical and useful quenching temperatures which are certainly not over 1650 degrees Fahr. Secondly this assumption, as far as we are aware, is most generally interpreted as applicable for practical and beneficial drawing times of 1 to 5 hours. Perhaps 10 hours would be more cautious and certainly is most inclusive. The authors' own data show a linear relationship up to the Ac_1 of all steels tested for tempering times of 1 to 5 hour durations. This is in accord with many data we have on straight carbon steels of 0.70 to 1.20 per cent carbon. The authors thus in fact prove that the assumption is justified for all types of straight carbon steels for "at heat" times up to somewhere between 5 and 25 hours and for some types up to 125 hours.

On two occasions the higher nickel content (0.14 per cent) of Steel F is suggested as accounting for its anomalous resistivity data. This is suggested on the basis of its retarding effect on the reaction rate when quenched. In view of the relative impotence of the element nickel on the rate of reaction and the fact that each of the steels contains some nickel, we cannot avoid feeling that any difference from this cause is purely imaginary. Specifically Steel C, with which Steel F is compared on one occasion, has 0.07 per cent nickel. Thus the difference in nickel is but 0.07 per cent. Steel C however has 0.11 per cent chromium while Steel F has 0.04 per cent. Since it is an established fact that chromium is far more effective (about triple) in its retarding effect on reaction rate than nickel, Steel F in light of these differences would have a faster, not slower, rate of reaction. However inasmuch as the several steels vary as much as 0.21 per cent manganese an element of still greater potency, a consideration of the small differences in either nickel or chromium is scarcely justified.

Though we were deliberately looking for a difference in the degree of carbide coalescence of all steels tempered 125 hours at 550 degrees Cent. (Fig.

17) the most outstanding feature appears to be the strong resemblance. However, Figs. 18 and 19 of Steels B and F respectively tempered 125 hours at 650 degrees Cent. do clearly show a contrast not only in distribution but in number of carbides. According to the fracture grain size data of Tables VI and VII, Steel B is coarse and Steel F is fine. We consider only the 1830 degrees Fahr. quench rating as pertinent here in view of the authors' use of this temperature in their enquiry since the only significant austenitic grain size is that established immediately prior to quenching. The authors thus have shown that Steel B having a very coarse fracture grain size (3) exhibits a distinctly slower rate of carbide coalescence and graphitization than Steel F having a moderately coarse fracture grain size (4½-5). The contrast by grain size seems to end here. Steel C whose reaction rate in tempering classifies it with Steel B (coarse) is second only to Steel F in fineness of grain. Moreover Steel E also grouped with Steel B (coarse) in tempering reaction rate must be placed among the fine grain steels on the basis of its microstructure and hardening reaction rate.

Authors' Reply

By B. S. Norris

The authors appreciate the comments submitted by Mr. Rose and they will endeavor to answer the questions which have arisen.

It is difficult to say how much the hardness is modified by small quantities of graphite since the size and distribution of the carbides are never constant during a given change in graphite particle size. A comparison between the hardness-temperature curves of Fig. 14 with the microstructures in Fig. 25 tends to show that the graphite particles must exceed a certain size and number before their presence exerts an effect on the hardness that can be distinguished from that produced by the coalescence of the carbides. Take for example, Steel F in Fig. 25. In this steel the graphite particles remain small up to at least 25 hours when tempered at 650 degrees Cent. This correlates with the linear curve shown in Fig. 14. However, many large graphite particles are present after the 75 and 125 hour tempering periods, and it is at these times that the "V" shaped curves appear. After 125 hours, Steel B also had a few medium sized graphite particles. Even these few seemed to be exerting a slight effect upon the hardness as is shown by the 125-hour curve of Steel B in Fig. 14.

Mr. Rose objects to our comments on the usual assumption that "decrease in hardness increases with tempering temperature up to the critical." No doubt this condition holds for the steels used in our research when considering short tempering times. However, we believe that the data obtained from prolonged periods of tempering are new and were not to be anticipated. More recent tests conducted on temperature gradient specimens have shown similarly surprising differences in the tempering behavior of the "similar" steels when hardened from 850 degrees Cent., which is in the commercial heat treating range.

It is not surprising that Mr. Rose failed to find a difference in the degree of coalescence in the carbide in Fig. 17. This is probably due to the poor reproduction. The original pictures of the microstructures, however, clearly reveal a difference. The difference is particularly evident between Steels "B"

and "F" which were the most and the least resistant to graphitization, respectively, of any of the steels subjected to tempering at 550 degrees Cent. which is shown in Fig. 17.

Since the preprinting of the paper the authors have obtained data indicating the amount of graphitization which occurred in the resistivity-coercive force bars on quenching from 1000 degrees Cent. and subsequently tempering for 125 hours at 550, 650 or 710 degrees Cent. We are indebted to A. C. Chamberlin, Bethlehem Steel Corporation, for the following analyses for graphite carbon.

	Tempering Temperature		
Steel	**550° C**	**650° C**	**710°C**
A	0.58	0.87	——
	0.58	0.89	0.55
B	0.01	0.74	0.15
	0.01	0.76	0.20
C	0.03	0.84	0.05
	0.05	0.80	0.09
D	0.43	0.70	0.41
	0.40	——	0.30
E	0.02	0.43	——
	0.03	0.46	0.03
F	0.58	0.86	0.60
	0.55	0.86	0.55

The data clearly indicate that graphitization is a maximum at 650 degrees Cent. However, the difference in graphite analyses between the various steels for a common treatment is less marked in these samples. The repeated heating and cooling to which the resistivity samples were subjected during the 125 hours tempering appears to promote more marked graphitization in those steels which are not nearly so prone to exhibit carbon dissociation when continuously annealed at temperature for the entire 125 hours.

The quantity of graphite indicated in the tabulated data may be observed to correlate closely with the behavior of the steels as revealed by the changes in physical properties discussed and recorded in the paper.

CREEP OF SOLID CYLINDERS IN TORSION

By M. C. Fetzer

Abstract

This paper considers the problem of torsional creep of solid cylinders. Short time tests, up to thirty-five hours, were made on 0.10 and 0.25 per cent carbon steels in several conditions. Creep rates were measured during the third to sixth, fifth to tenth and twenty-fifth to thirty-fifth hours by the method of Pomp and co-workers. An attempt was then made to correlate creep rates in torsion with creep rates in tension using for the latter data from Pomp and Höger on similar steels. To make such a comparison it was necessary to approximate the stress distribution in solid cylindrical torsional specimens. This approximation indicated that the maximum stress during creep in torsion was seven per cent greater than if the stress were uniform throughout the section. The comparisons showed that the shear creep rate in tension is faster than in torsion for the same shear stress, and it is suggested that this is due to greater availability of slip planes in tension.

ALTHOUGH much interesting and valuable information may be obtained from torsional creep tests, the greater use of materials in tension has relegated this method of testing to the background. Literature relating to the subject of torsional creep is not abundant. Experimental work has been carried out by P. Henry (1)[1], R. W. Bailey (2) (12), F. L. Everett (3), G. R. Brophy (4), and F. C. Lea and C. F. Parker (5). Very few long time tests of practical value have been carried out. The author has found the work of R. W. Bailey to be especially helpful in the preparation of this paper.

Solid Versus Hollow Twist Specimens

Why, as the title states, were solid twist specimens used instead of hollow ones insomuch as the stress conditions in solid specimens

[1]The figures appearing in parentheses refer to the bibliography appended to this paper.

The author, M. C. Fetzer, is instructor in metallurgy, Pennsylvania State College, State College, Pa. Manuscript received May 1, 1936.

become complex with plastic deformation whereas in hollow ones they do not. The reasons can be given as follows: (a) If it is desired to determine the elastic limit, proportional limit, yield point, or maximum strength of a solid cylinder resulting from twist, these values cannot be obtained from hollow cylinders. Even though deformation is entirely elastic and the stress therefore known, it is usually found that a greater stress is required to cause a low carbon steel to yield in the solid than in the hollow cylinder. If it is impossible to convert from one to the other in this case it is to be expected that the maximum stresses necessary to produce the same creep rates will also differ. Usually service requires solid parts; it is, therefore, desirable to test solid parts. (b) The stress conditions in a solid specimen may be complex, but as Bailey has shown and as will be discussed later in this paper the maximum stress can be determined with some degree of accuracy. The fact that it cannot be exactly determined should not condemn it to uselessness. Bailey says: "In the case of solid circular shafts, as the shear stress will not vary greatly with the radius, it will be sufficiently accurate to calculate the shear stress f′ for a torque T by the following relation which applies to the case of constant shear stress

$$f' = \frac{12T''}{\pi d^3} \quad \ldots\ldots\ldots\ldots (2)$$

(c) In many cases it would be difficult and expensive to form a hollow cylinder for test and the specimen would in some instances not be representative of the whole. (d) A solid torsional specimen has the advantage over tubular type specimens in that it does not buckle after slight strain nor does it present the possibility of free surface effects not ordinarily encountered in most practical applications.

Everett has shown that the beginning of collapse in tubular specimens is accompanied by an apparent increase in creep rate. Tensile specimens also show an increased creep rate in the so-called "third stage"; the reason for which is not completely explained. On the other hand, none of the tests presented here exhibited an increase in creep rate with time. This may be due to no appreciable dimensional change altering the stress such as occurs in tensile specimens undergoing strain. It is not a question of deciding upon one test specimen or the other as a standard—each has its field, each is differ-

ent—at least until more is known regarding the mechanics of failure both elastic and plastic.

In this paper the results of some tests on 0.10 and 0.25 per cent carbon steels are given; this is preceded by a description of the testing equipment and procedure employed. A method of determining the maximum stress in the test specimens is given and the torsional results of these steels are compared with those of similar steels in tension obtained from the results of other investigators. All of the creep tests were of short duration.

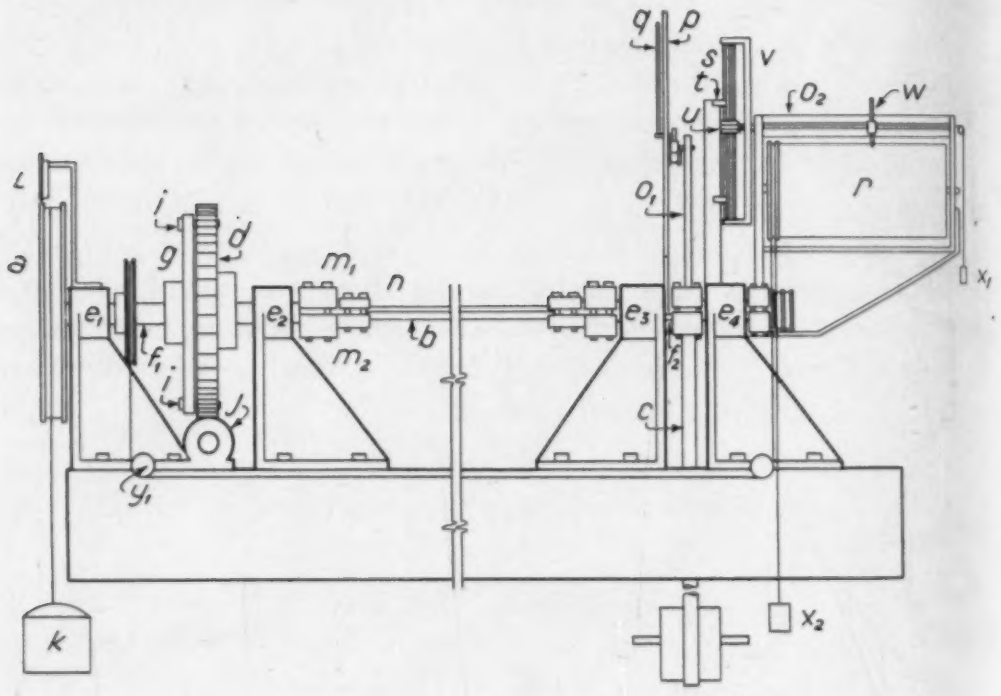


Fig. 1—Diagram of the Sauveur Torsion Creep Machine.

Torsional Creep Testing Apparatus and Testing Procedure Used

Testing Machine Proper: The machine used in this investigation was suggested by Professor Albert Sauveur and built by the Baldwin-Southwark Company. It is a combination torsional and torsional creep testing machine built so that torque can be applied with a motor or by means of dead weights. The machine, which has a 1000 inch-pounds capacity, is shown diagrammatically in Fig. 1. When used for creep testing the pendulum shown is fixed and loading is applied with dead weights. Fig. 2 is a general view of the

machine equipped for creep testing. The two ball bearings E_1 and E_2 (Fig. 1) minimize bending of the specimen upwards when load K is applied. Friction is made almost negligible by removing gear D and plate G as the motor drive is not used for creep. Tests showed that with 120 inch-pounds on the specimen, the friction was less than 0.4 per cent.

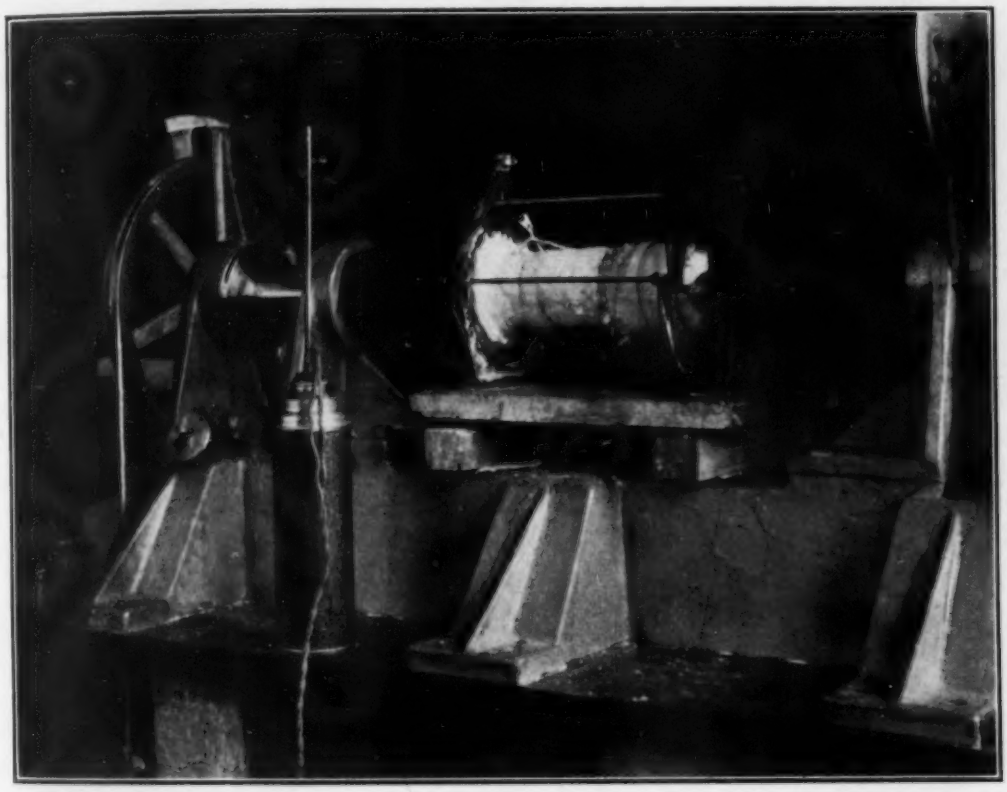


Fig. 2—General View of Sauveur Torsional Machine Equipped for Creep Testing.

Furnace and Temperature Control: The furnace and temperature control was built by F. H. Norton and J. A. Fellows at the Massachusetts Institute of Technology. A vertical furnace similar to this one used for tensile creep tests has been described by them in a recent publication (6). Fig. 3 is a diagrammatic sketch of the controlled furnace. Essentially it consists of an 18-8 steel tube A (1.50 inches I.D., 1.75 inches O.D., and 11 inches long). This is wound with mica tape on top of which is wound the heating coil of No. 18 chromel wire. There are 37 turns as follows: first inch at left 12 turns, next 2 inches has 8 turns, next 4 inches has no turns, next 2 inches has 7 turns, and last inch 10 turns.

The temperature is controlled automatically by the movement of the furnace tube as follows: The tube attached rigidly to the cast

iron plate M expands on heating. Attached to the tube is the casting B, which, when the tube expands, causes the semi-circular casting D to pivot about point O by means of the steel strips E and E'. Attached to casting D is the lever F which is thrown downward by this pivoting thereby breaking the auxiliary circuit at the contact. When the contact is broken the lever is displaced upwards again and

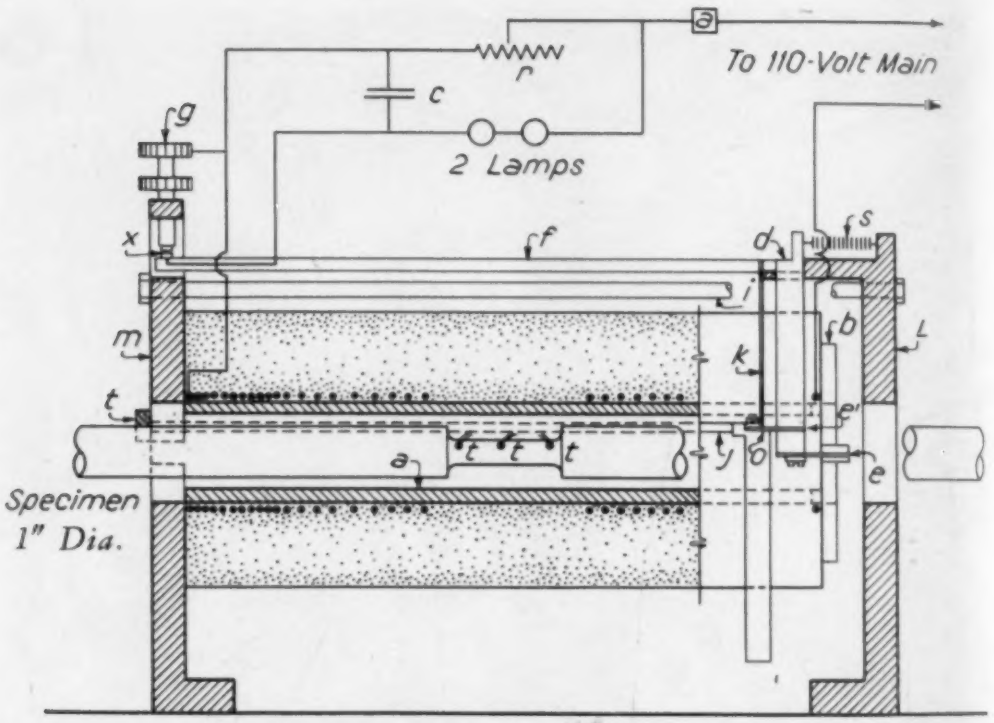


Fig. 3—Diagrammatic Sketch of the Controlled Furnace.

closes the circuit. Lever tube F is kept taut by the spring S. The whole furnace tube and insulation is supported like a cantilever beam except for two thin brass strips K attached to casting L which aid in suspension. Fine adjustments of temperatures are made by turning screw G (1 turn is about 5 degrees Cent.); coarse adjustments are made with nut T, which pulls invar rods J. The furnace is supported by invar rods I which, because of their low expansion, minimize changes in furnace temperature due to room temperature changes.

Included in Fig. 3 is the wiring diagram for controlling the heating current. The variable resistance R which controls the main current is composed of three rheostats in series (3.1 amperes, 43 ohms; 4.4 amperes, 21.5 ohms; and 6.2 amperes, 1.1 ohms). When contact X closes about 10 per cent additional current is allowed to

pass through the two lamps which are 200 watts each. A condenser C reduces sparking as contact X makes and breaks. An ammeter (12 amperes) is introduced in the circuit to assist in obtaining the desired temperature. Tests at 400 degrees Cent. (750 degrees Fahr.) take about 5 to 5.5 amperes. The temperature over the 2-inch gage length is quite uniform; the maximum difference being about 3 degrees Cent. It is quite a simple matter to take out windings or to put windings in by digging through the insulation to the wires. The control keeps the temperature constant to within a few tenths of a degree Cent. if the room temperature remains constant. The furnace, temperature variations, uniformity, etc., are within the standards set up by the A.S.T.M. for creep tests.

Test Specimens: The creep test specimen used is shown in Fig. 4. Although a gage length of only 2 inches was used because

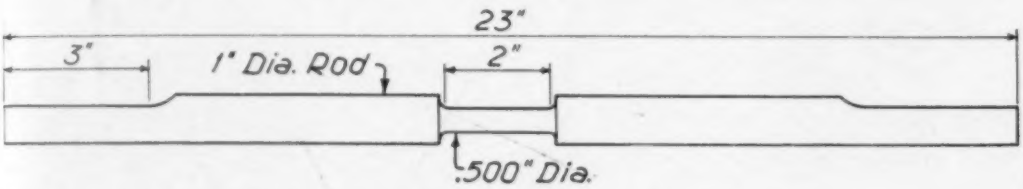


Fig. 4—Sketch of Creep Test Specimen Used in These Tests.

of the relatively high displacements measured, it would perhaps be possible to use a gage length up to 4 inches and still keep within the A.S.T.M. specifications. Utilizing a specimen shank 1 inch in diameter, a gage diameter of 0.5 inch is small enough to permit only a small amount of creep in the shank. All specimens were machined from 1⅛-inch rounds so as to allow sufficient material to permit machining the entire length in order to remove warpage effects due to heat treatment and thereby keep the gage length centered in the axis of twist.

The specimens fit into chucks at either end. A flat section is milled off both ends of the specimen longitudinally for about 3 inches which permit the specimen to be bolted at either end to the chuck by means of two bolts and a plate. This arrangement of gripping, although it requires a specimen about 23 inches long, eliminates slipping. No bending of the specimen has ever been noticed from the time of insertion in the creep furnace until removal (about 3 days).

Temperature Measurements: Three chromel-alumel thermocouples were attached to each specimen, one in the center and one at

each end of the gage length (Fig. 3). The couples were tied to the specimen at two locations along the 1-inch section with asbestos string and the beads were fastened to their proper locations at the gage length with fine invar wire. The same couples were used on all tests and these showed the temperature variation along the gage length to be not more than 3 degrees Cent. A Wolff type potentiometer reading to 0.014 degree Cent. was used. It was found that the room temperature varied not more than 2 or 3 degrees Cent. during a test and the furnace temperature did not fluctuate over a few tenths of a degree Cent.

Measurement of Creep: The device for measuring creep is shown in Fig. 5. Advantage is taken of the relatively large movement near the periphery of the torque wheel as compared with that at the periphery of the gage diameter as essentially measurements are made of movements of the periphery of the wheel under high magnification.

To magnify the movements of the wheel an ordinary desk microscope was fastened rigidly to the base of the testing machine and sighted at a reference mark on the periphery. This mark was allowed to play on a drum covered with bromide paper which turned once a day so that creep was recorded laterally on the drum and time along the circumference.

In order to get a satisfactory curve it was necessary to have a reference mark which would still be quite fine even after magnifying about 100 diameters. A satisfactory arrangement was reached with needles mounted on a brass plate. This plate was placed in line with the microscope on the periphery of the wheel and a beam of light played upon it. The beam when adjusted correctly caused a narrow bright line to be reflected from each of the small needles. This vertical line was then reduced to a point by means of a narrow slit. The result was a small spot of light playing on the bromide paper. The camera with the drum inside is shown in place in Fig. 6.

Method of Running a Test

The tests were all of short duration, usually about 36 hours; the total time for a test was 3 days although with slightly more vigilance a test could be run in 2 days. The procedure was as follows: Before tightening up the specimen in the chucks it is necessary to adjust the system to allow for longitudinal thermal expansion of

the specimen as this amounts to nearly a tenth of an inch up to 400 degrees Cent. (750 degrees Fahr.). There is sufficient longitudinal play in the machine to care for this. After the specimen had been

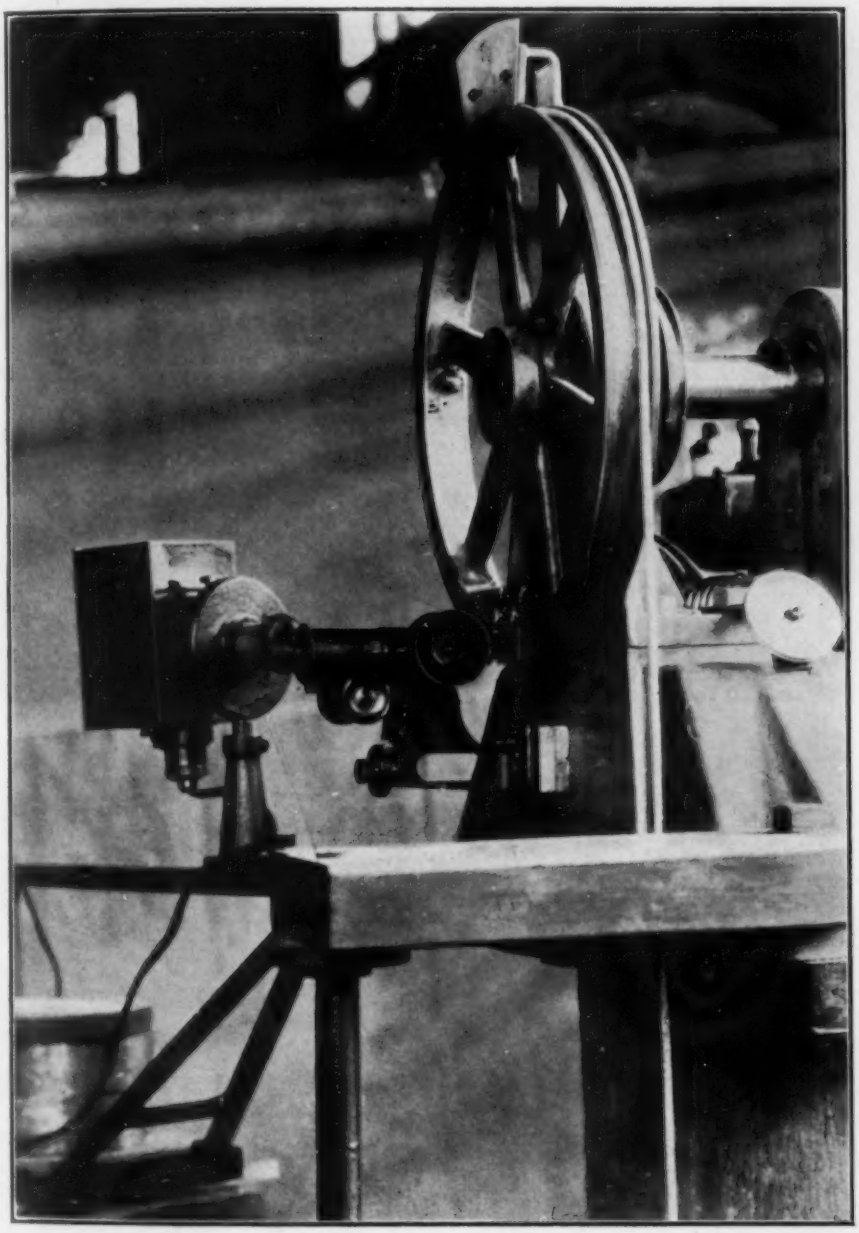


Fig. 5—Photograph of Device for Measuring Creep.

fastened in the chucks the latter were wrapped with felt; this precaution minimizes cooling of the chucks by occasional drafts thus helping to maintain a uniform temperature. At this point the arrangement is as shown in Fig. 2.

The current was then applied; a temperature of 400 degrees

Cent. (750 degrees Fahr.) being attained in about 2 hours. It was customary to apply the test load the next day as the temperature tended to climb somewhat for about 24 hours. The temperatures of the three couples were then recorded and the vernier on the torque

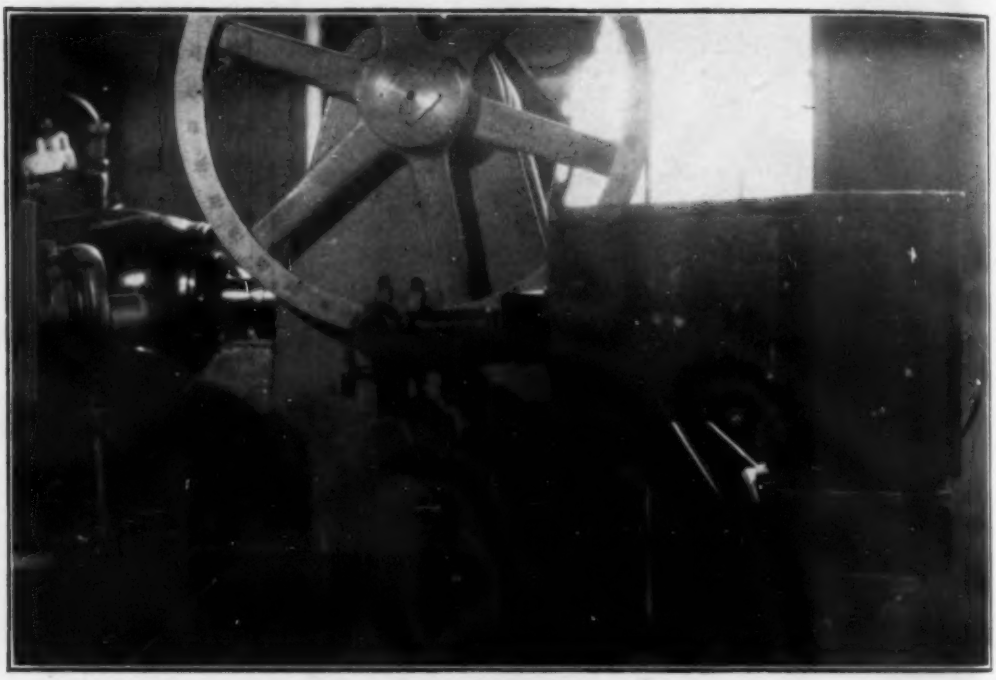
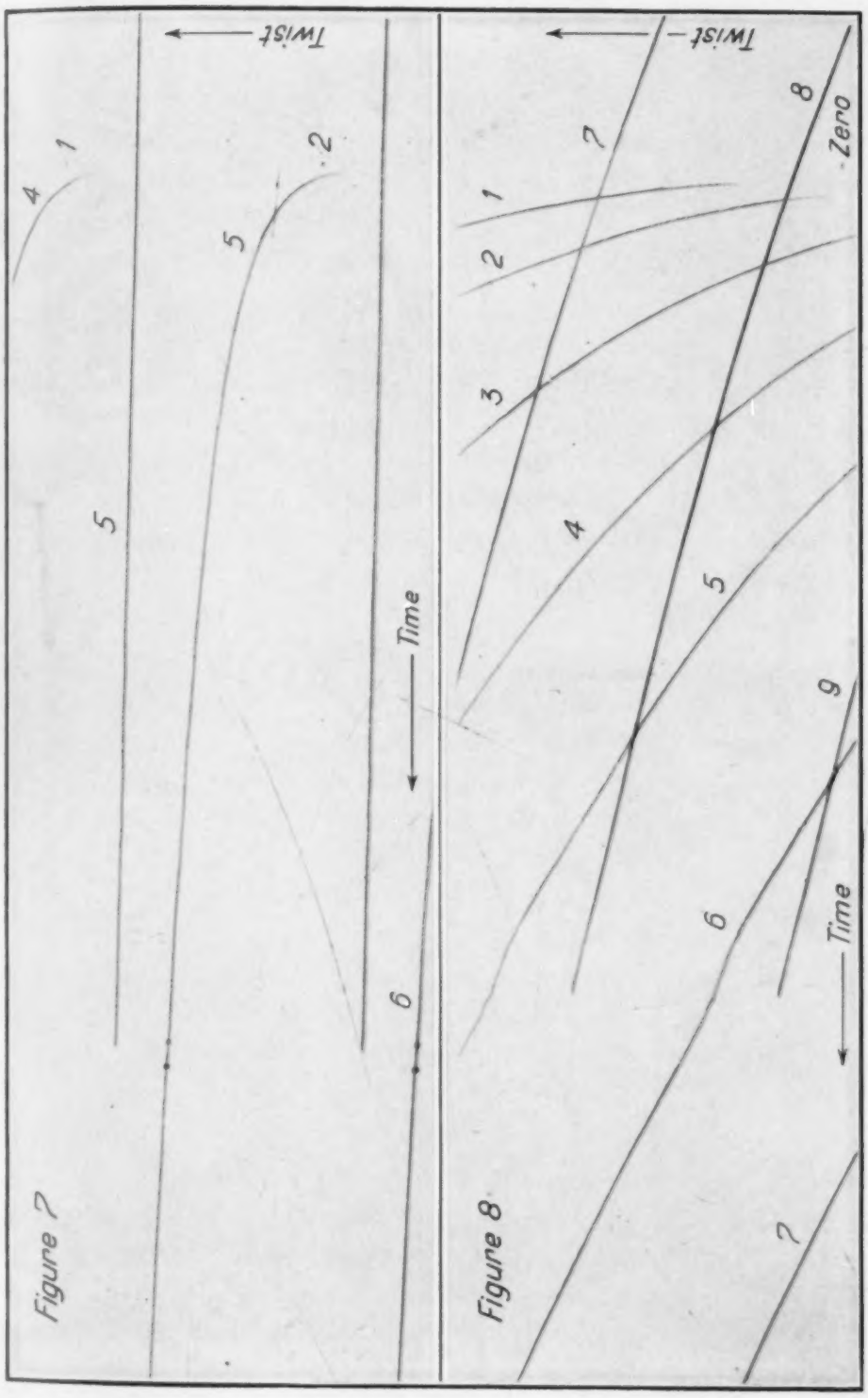
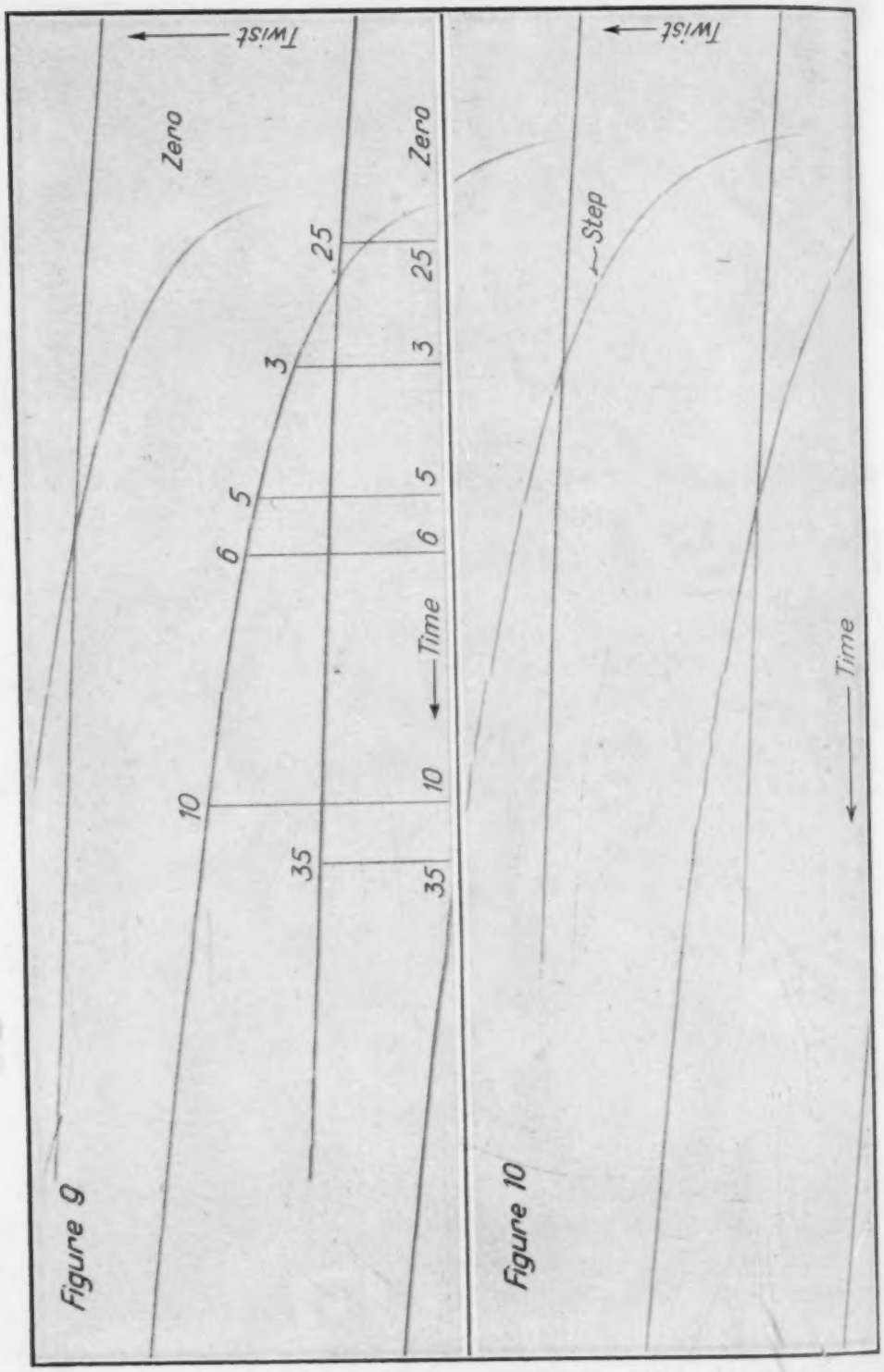


Fig. 6—Photograph Showing Camera in Place.

wheel read to the nearest 0.05 degree. The reflection of the needles was then focused on a ground glass and the camera put in place. Next, with no load on, the illumination was applied to the needles and a zero load mark made on the bromide paper. The desired load was then applied slowly by pouring lead shot into the bucket attached to the torque wheel. As soon as possible after adding the shot a second reading of the vernier was made. In no case was the application of the load found to alter the readings of the thermocouples to any marked degree. The next day, after the load had been on for about 12 to 15 hours, the temperatures of the three couples were again recorded. The following day, after the load had been on for 35 hours or more, the temperatures were again recorded and the vernier read. After removing the camera the load was removed and the vernier again read. This finished the test and another specimen could be inserted at that time. Typical curves obtained in this manner are shown in Figs. 7 to 10. Portions of the curves are in duplicate due to more than one needle reflection.



Figs. 7 and 8—Photograph of Typical Curves Obtained on Creep Testing Machine.



Figs. 9 and 10—Photograph of Typical Curves Obtained on Creep Testing Machine.

Factors Determining the Sensitivity of the Measurements

The following factors, exclusive of the size of the specimen, determine the sensitivity: diameter of the torque wheel; magnification of objective plus ocular; distance from paper to ocular; smallest measurable distance on the curve.

The torque wheel is 16 inches in diameter or 32 times that of the specimen while the smallest displacement measured from the curves was 0.01 inch. The distance from the ocular to the paper is about 10 inches. Any desirable combination of ocular and objective up to 250 diameters could be used for recording and up to 1000 diameters for visual observations. Other factors determining the sensitivity of measurements exclusive of the kind of equipment are: gage length and diameter; time of test.

Advantages: The device just described has many advantages; the greatest of these are, first, that the system is 100 per cent optical so that the measuring device does not interfere in any way with the creep machine proper; and second, any amount of twist can be measured automatically. In regard to this latter feature, as the spot of light from one needle leaves the paper another appears. Usually there are spots from two needles on the paper so that duplicate curves are recorded. If it were desired to measure creep up to one complete revolution or more it would be necessary to attach say 720 needles to the periphery of the wheel, i.e., one at each half degree. The author is not aware of any other simple optical creep device which gives a continuous curve for large angles of twist. Another advantage is that the strain device is not influenced by the temperature of the test. Objections from this source are, however, not as serious in torsion as they are in tension.

The device is simple and rugged; about 30 curves have been run and every one has been satisfactory. The whole device is compact and built integral with the machine so that there are no vibrations of one part which do not affect the others. Furthermore, as will be discussed, the system is very sensitive.

One objection to this system of creep measurement might be that the movement measured is that from the whole specimen and not merely that from the gage length. This disadvantage is not, however, as bad as it might at first seem. The bar shank is twice the diameter of the reduced section which means the stress is about

1/7* as much and when it is considered that creep varies as some power of the stress (perhaps the 6th power) this external creep becomes very small even though the extraneous metal is 8 times as long as the gage length. It is, however, advisable to run a test on a specimen having no reduced section so as to determine approximately how much creep is due to flow outside the gage length. Such a test indicated that this creep amounted to about 7 per cent of that taking place in a standard specimen. This value was adopted for correcting creep rates to determine the most probable rates in the tests to be described.

Sensitivity: The sensitivity with which the shear strain can be measured depends on the magnification used. For example with the creep specimen described and measuring to the nearest 0.01 inch on the curve the following strain sensitivities can be had:

× 55	2.84×10^{-6} in./in.
× 250	6.25×10^{-7} in./in.
× 1000	1.56×10^{-7} in./in.

For all of the creep tests to be described a magnification of × 55 obtained by means of a 16 mm objective and a × 7.5 ocular was employed.

The sensitivity at × 1000 is of the same order of magnitude as that obtained by Everett (3) who states that the sensitivities he obtained were greater than those found in any laboratory.

The torque wheel vernier L (Fig. 1) reads to 0.05 degree which means that shear strains of 1.1×10^{-4} inch per inch can be detected by this means when using the specimen described.

Precision: There are factors other than the sensitivity of the strain measuring equipment which affect the accuracy of creep rate measurements. For example furnace temperature changes and fluctuations cause oscillations of a loaded twist specimen in an analogous manner to which a tensile specimen elongates and contracts with temperature changes. Thus Everett (3) has pointed out that theoretically these temperature changes cause changes in the modulus of rigidity, polar moment of inertia, and length. Practically, however, the torsional creep curves showed no such oscillations except for a few time intervals. On the other hand these changes are very distinct in autographic tensile curves. The usually smooth nature of the torsional curves makes it possible to detect any irregu-

*Actually between 1/6 and 1/8 depending on the amount of plastic strain.

larities in the creep process. In tensile curves these irregularities are obscured.

Materials Tested

Twenty-five creep tests of three days total time each were made. For these tests three different supplies of steel were used, but two of these (Nos. 10 and 20) appear to have come from the same heat as they analyzed nearly alike; they were both purchased from the same company. The analyses of these steels are:

Designation	Carbon Per Cent	Manganese Per Cent	Phosphorus Per Cent	Sulphur Per Cent	Silicon Per Cent
20	0.26	0.54	0.006	0.033	0.03
10	0.25	0.54	0.007	0.025	0.04
5	0.10	0.50	0.025	0.065	0.02

A 22-foot length of each of these, 1⅛-inch round hot-rolled, was purchased and cut into test specimen lengths of about 22 inches each. All of the test specimens from steel 20 were given a "stabilizing" treatment which consisted of first annealing by furnace cooling from 900 degrees Cent. (1650 degrees Fahr.) after holding for 1 hour at that temperature; and then reheating to 600 to 650 degrees Cent. (1110 to 1200 degrees Fahr.) and holding in that range 4 or 5 hours. The reason for giving the steel this treatment was to put it in such a stable condition that 40 hours at the creep testing temperatures of 300 to 500 degrees Cent. (570 to 930 degrees Fahr.) would not further alter the creep characteristics to any marked degree. This stabilizing treatment did, incidentally, have a marked effect in lowering the creep resistance over the as-received or normalized conditions. Tests were made on this steel at 350, 400, and 500 degrees Cent. (660, 750, and 930 degrees Fahr.) for times of up to 40 hours and in one case for 87 hours at 400 degrees Cent. (750 degrees Fahr.). Steel No. 5 was tested in the as-received, normalized, and "stabilized" states. All machining was done after heat treatment in order to avoid decarburization.

Test Procedure of Pomp and Co-workers

The results of these torsional tests were compared with tensile data of Pomp and his co-workers (7, 8, 9, and 10). Their heating medium was a salt bath; about 4 hours elapsed from insertion of

specimen until loading. Their creep curves were recorded continuously and automatically by a photographic method. They then measured the average creep rates during the third to sixth, fifth to tenth, and twenty-fifth to thirty-fifth hours and expressed it in terms of 10^{-4} per cent per hour. Likewise, the creep rates of the present investigation were measured during the same time intervals in order to permit comparisons. Some of the steels tested by these investigators were similar in composition, structure, and physical properties to those tested here while at the same time their steels varied from one to another in a degree equal to any differences between their steels and those presented here. Data on the steels compared are given in Table II. Undoubtedly the best method of comparing creep data of this sort is to test the same materials in the same laboratory in both tension and torsion but unfortunately this was not found feasible. The torsional creep test results are given in Table I.

Discussion of the Creep Curves and Data

Interpretation of the Autographic Curves: All creep curves were recorded in the continuous manner already described; four typical curves are shown in Figs. 7 to 10. The original curves are 13 inches long; upon which one degree of twist produces a vertical movement of about 7.7 inches. To explain Fig. 7, for example, which is from specimen 5-II and shows relatively slow creep, let us consider successive needle reflections to be numbered 1, 2, 3, etc. Spots 1 and 2 are starting reference marks from needles 1 and 2 obtained with no load and 5 minutes time. These spots represent zero twist and time. Upon applying the load (24.2 pounds—Table I) the specimen instantly twisted 0.70 degree which is a vertical movement of about 5.4 inches on the original curve. By this twist needles 1, 2, and 3 were displaced upwards and off the paper and needles 4 and 5 commenced to record as shown. The time-deformation curve was then recorded in duplicate until curve 4 left the paper after about 2 hours. Until the eighth hour curve 5 alone recorded when curve 6 appeared as the reflection of needle 6. From the eighth hour until the finish of the test the curve was again recorded in duplicate. It is only with very small rates of creep such as this that one needle registers a complete curve over the whole time as needle 5 has done in this case.

Going to the other extreme, Fig. 8 shows the curve of specimen 5-VII which exhibited a high rate of creep. Here, in addition to the

Table I
Data Relative to Creep Tests

Spec. No.	% C	Treatment	Pre-heat Hrs.	Actual Load lbs. at 8 ins.	Stress lbs./in.² Uniform Stress	Stress lbs./in.² 1.07 x Uniform Stress	Temp. Deg. C.	Total Strain in/in. x 10^3	Recovered Strain in/in. x 10^3	Total Plastic Strain in/in. x 10^3	Total Time Hrs.	Creep Rates in 10^{-4} %/hr. in the following hrs. 3-6	5-10	25-35
20-XII	0.26	S*	24	34.2	8,370	8,960	350	4.11	1.75	2.37	40	41	29	9.9
20-VIII	0.26	S	8	34.2	8,370	8,960	353	4.52	1.85	2.68	37	50	37	12.2
20-IX	0.26	S	20**	34.2	8,370	8,960	350	4.57	1.92	2.66	39	44	31	12.4
20-I	0.26	S	9**	24.2	5,930	6,350	399	2.91	1.45	1.46	39	45	32	12.4
20-VII	0.26	S	6	24.2	5,930	6,350	399	2.47	1.03	1.44	38	40	29	10.3
20-X	0.26	S	7	24.2	5,930	6,350	396	2.86	1.23	1.63	87	36	26	10.5
20-II	0.26	S	24**	27.2	6,660	7,120	399	3.67	1.51	2.16	37	59	44	18.3
20-XIII	0.26	S	116***	30.2	7,400	7,910	395	3.06	1.63	1.43	36	43	33	13.6
20-VI	0.26	S	30	39.2	9,600	10,280	399	14.08	2.10	11.98	37	219	166	56
20-V	0.26	S	30	10.2	2,500	2,680	499	1.79	0.63	1.16	38	38	28	14.8
20-III	0.26	S	30	10.2	2,500	2,680	500	1.78	0.67	1.11	36	39	30	14.8
5-II	0.10	A.R.	27	24.2	5,930	6,350	400	2.08	1.38	0.70	38	18.8	12.0	3.4
5-XII	0.10	A.R.	30	31.2	7,650	8,190	400	3.33	1.71	1.62	35	37	26	8.6
5-V	0.10	A.R.	31	36.2	8,860	9,410	400	3.62	1.91	1.71	37	77	52	18.1
5-XI	0.10	N	30	31.2	7,650	8,190	399	3.60	1.75	1.85	40	40	25	8.0
5-VII	0.10	N	30	41.2	10,100	10,800	398	10.30	2.16	8.14	37	160	107	38
5-X	0.10	A.R.	28	70.2	17,200	18,400	302	35.20	3.85	31.35	..	29	18	4.7
5-IX	0.10	S	30	36.2	8,860	9,410	400	9.40	2.07	7.33	38	130	81	27
5-VI	0.10	N	30	25.2	6,160	6,590	500	21.70	1.78	19.92	39	780	610	..
10-V	0.25	A.R.	56**	39.2	9,600	10,280	400	4.61	2.15	2.46	37	49	35	10.4
10-II	0.25	S	31	39.2	9,600	10,280	400	17.43	2.24	15.19	17	420	310	..

*(S) stabilized; (N) normalized; and (A.R.) as-received.
**One cooling.
***Three coolings.

two needles giving the reference points, nine needles have contributed to the record. The portion of the curve recorded by needle 1 is scarcely visible; the faintness is due to creep occurring at such a fast rate that the exposure on the photographic paper is insufficient. To

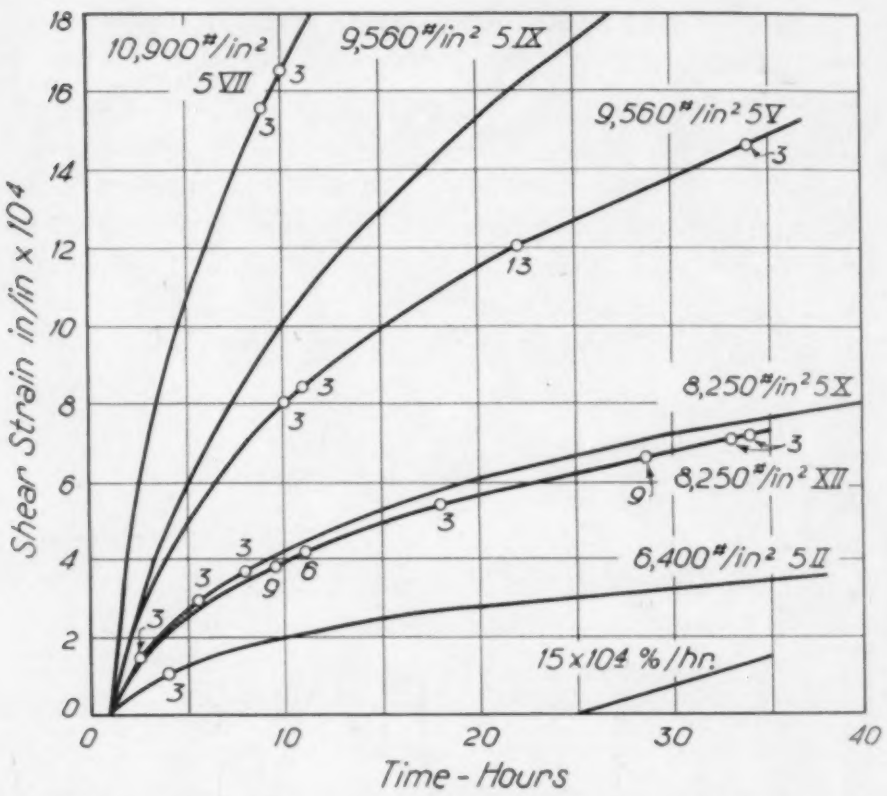


Fig. 11—Creep Curves of 0.10 Per Cent Carbon Steel at 400 Degrees Cent. (750 Degrees Fahr.).

interpret Fig. 8 one follows curve 1 upwards until it leaves the paper then traces vertically downward until striking curve 2 which in turn is followed until it leaves the paper and so forth until the end.

All of the creep curves from which the data given in Table I have been obtained were replotted to a more suitable scale and are shown in Figs. 11 to 15.

Calculation of Creep Rates: To give an example of how creep rates were determined* Fig. 9 showing curve 5-XI is included. To determine the creep rate in the fifth to tenth-hour period for example the height 5-5 in inches is subtracted from the height 10-10 in inches and the calculation made as follows, using actual figures obtained in laboratory tests.

*This is the method used by Pomp and his co-workers.

If

$\textcircled{1}$ = creep rate
l = length of gage = 2.12 ins.
t = time interval = 5 hrs.
X = magnification = 55 X
d = recorded creep movement in inches for time interval t (d = 0.50 in.)

$$\textcircled{1} = \frac{0.50}{55 \times 32 \times 2.12 \times 5} = 27 \times 10^{-6} \text{ in./in./hr.} = 27 \times 10^{-4} \text{ per cent per hour}$$

where 32 is the ratio of torque wheel radius (points of reflection) to specimen radius. The rate 27 x 10^{-4} per cent per hour represents

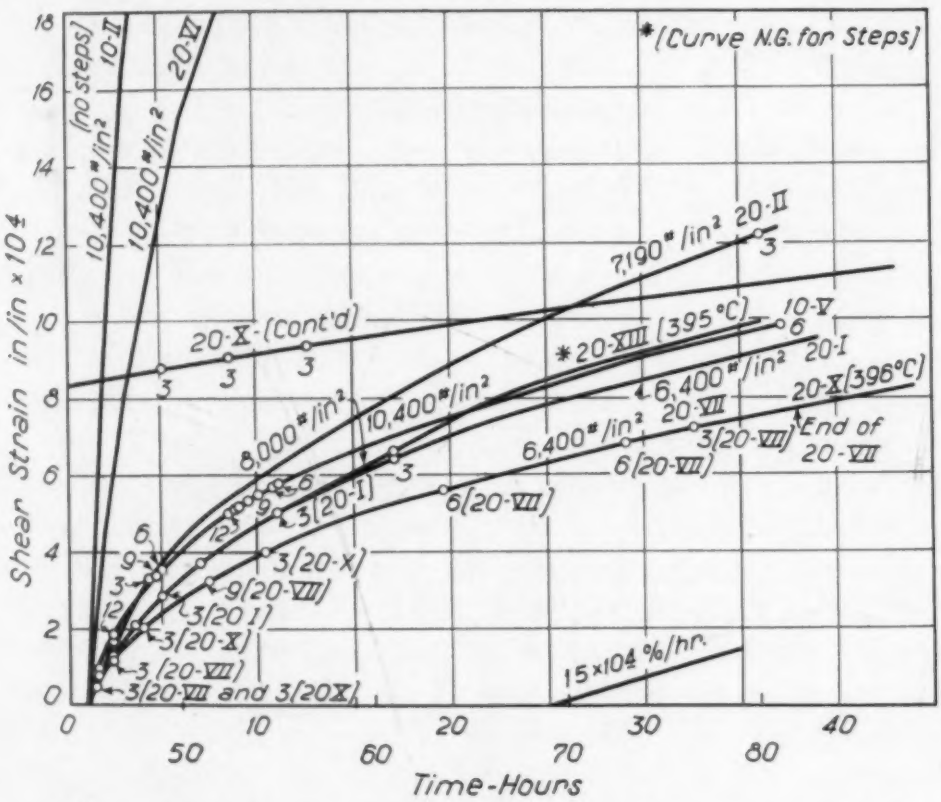


Fig. 12—Creep Curves of 0.26 Per Cent Carbon Steel at 400 Degrees Cent. (750 Degrees Fahr.).

the creep of the whole length; the creep of the gage length is 93 per cent of this or about 25 x 10^{-4} per cent per hour and it is this last figure which is given in Table I. In a similar manner the creep rates for the time intervals of third to sixth hours and twenty-fifth to thirty-fifth hours are determined.

Steps or Aging: Nearly all of the curves showed that creep was discontinuous; apparently flow occurred smoothly for a time and then a step occurred. One of the best examples of steps was shown

by an as-received specimen of 0.25 per cent carbon steel, 10-V (Fig. 10). These steps usually are represented by displacements of from 0.01 to 0.04 or 0.05 inch or 3 to 15 x 10^{-4} per cent. Perhaps with greater sensitivity it would be found that the apparently smooth portions of the curve are also discontinuous. It will be noted that the creep rate is slowed up for a period of time immediately following each step. The small numbers along the replotted creep curves (Figs. 11 to 15) indicate that steps occurred at that time and give the magnitude of the step in 10^{-4} per cent. It is possible that this stepping phenomenon can be associated with aging. In general it will be found that steps are more numerous the lower the temperature (compare Figs. 14 and 15). One test, 5-X, was run at 300 degrees Cent. (570 degrees Fahr.) as it was anticipated that at this lower temperature, nearer the blue heat range, more steps would result, and although many steps did occur the quantity and size of them did not meet expectations. It will be observed, however, that the creep curve of 5-X exhibits a slow rate and it is quite possible that much stepping took place prior to the time the movement became slow enough to record (first minute or less) as the deformation during this time was over 15 degrees. The creep during the remainder of the test was only about 1.6 degrees.

Another item of interest regarding steps and aging is shown by the normalized steel, curve 10-V (Fig. 12). This curve, exhibiting numerous steps, started out for the first few hours at a rate greater than that of the stabilized steel, 20-II, which was stressed at 7190 pounds per square inch but before 24 hours had passed was creeping at a rate about the same as that of the stabilized steel, 20-I, which was loaded to only 6400 pounds per square inch. Stepping may account for this slowing down of creep and we should expect the same from aging.

It would be difficult to attribute these steps to shocks because they appear to follow a law of appearing in greater number at the beginning of test than at the end and incidentally occur during the dead of night when shocks are at a minimum. The machine was mounted on concrete pillars on a concrete floor. It would be difficult with automatically recorded tensile creep curves to determine whether or not such steps occur, for thermal changes make the tensile curve saw-tooth like.

Total and Recovered Twist: Measurements were also made of

the total twist and the twist recovered on unloading. Unlike creep strain an appreciable portion of this twist is due to the elastic strain of the 1-inch section. These values are given in Columns 9 and 10, Table I. The values given for recovered twist were obtained from readings made immediately after unloading; it is known, however, that appreciable twist continues to be recovered for many hours after unloading, as illustrated in Fig. 13. Here, after 22 hours of

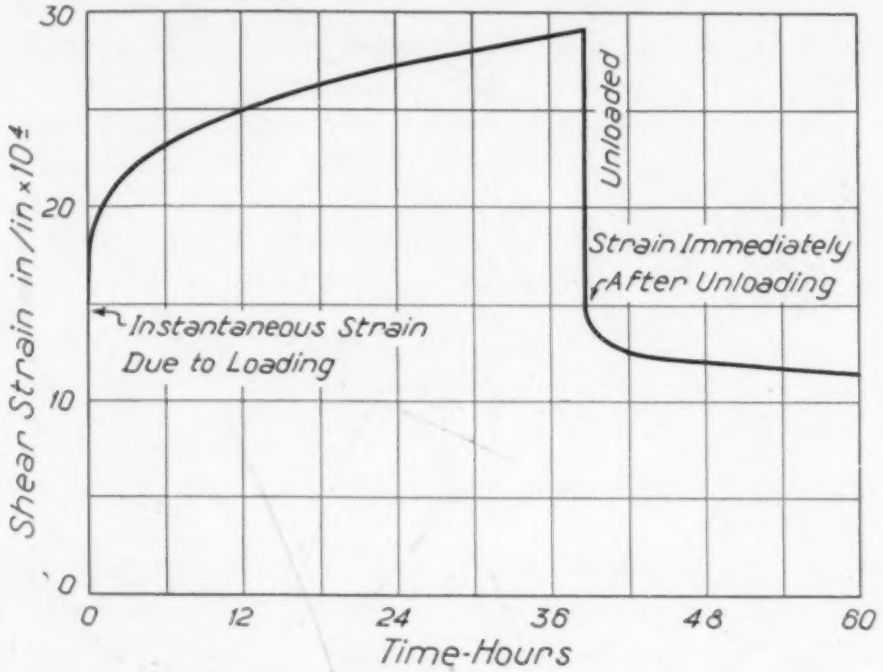


Fig. 13—Time-Strain Curve for 0.26 Per Cent Carbon Steel Showing Positive Creep Under Load and Negative Creep After Unloading. Torque—193.6 Inch-Pounds, Diameter Specimen—0.5 Inch, Temperature—400 Degrees Cent. (750 Degrees Fahr.).

relaxation, the additional recovered twist amounted to over 21 per cent of the instantaneous back twist and was still recovering at the rate of 2×10^{-4} per cent per hour. The total plastic twist values were obtained as the difference between the total twist and the elastic strain recovered. These values are given in Column 11, Table I.

Duplication of Creep Curves: Fig. 14 shows two separate tests on specimens 20-III and 20-V loaded to 2700 pounds per square inch at 500 degrees Cent. (930 degrees Fahr.). By taking 1 hour as zero creep the curves check very well. Curves 20-I, 20-X, and 20-VII (Fig. 12) also represent three separate tests run under nearly identical conditions, i.e., 6400 pounds per square inch and 400 degrees Cent. (750 degrees Fahr.); curves 20-VII and 20-X coincide while 20-I is somewhat higher. Curves 20-VIII, 20-IX, and 20-XII (Fig.

15) also represent conditions which differ but little. All of these comparative tests were made on steel in the "stabilized" condition, namely; normalized, then heated for 4 hours at 600 degrees Cent. (1110 degrees Fahr.) and furnace cooled.

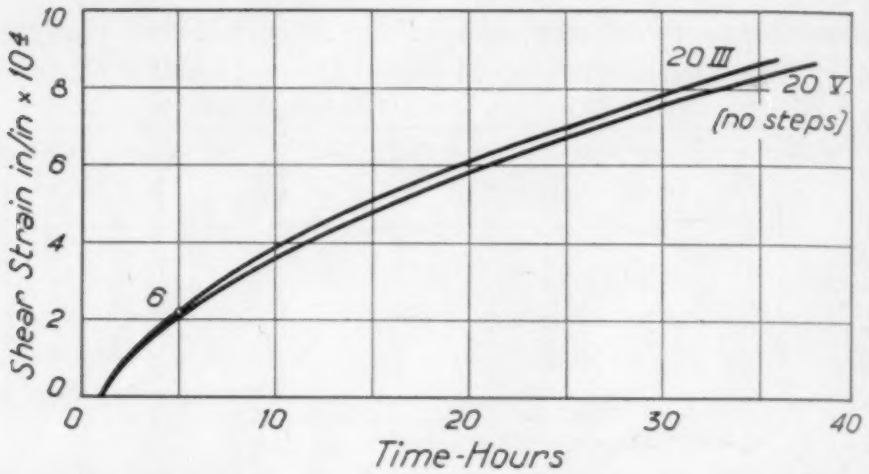


Fig. 14—Creep Curves in Duplicate Stabilized 0.26 Per Cent Carbon Steel at 500 Degrees Cent. (930 Degrees Fahr.) and 2700 Pounds Per Square Inch.

Effect of Various Heat Treatments on the Creep Curves

0.10 Per Cent Carbon Steel: The 0.10 per cent carbon steel (5) was tested in the as-rolled, normalized, and "stabilized" conditions. In addition to creep tests the microstructures were studied and Rockwell hardnesses determined. There is no apparent difference in the as-rolled and normalized steels (Fig. 16) but the "stabilizing" has divorced the pearlite somewhat (Fig. 17). The as-rolled specimen had a Rockwell of 60B and the "stabilized" 55B.

The results of the normalizing treatment on the creep properties are shown by the two curves, 5-XI and 5-XII (Fig. 11); the testing conditions were as nearly alike as possible. The normalized specimen runs a trifle lower than the as-rolled but the difference is well within the ability to duplicate.

The stabilized steel, on the other hand, showed a greatly reduced resistance to creep. Curves 5-IX and 5-V of the stabilized and as-rolled steels, respectively, illustrate this decreased resistance, the creep rates being 27 and 18 x 10^{-4} per cent per hour in the twenty-fifth to thirty-fifth hours. The greater number of discontinuities or steps shown by the as-rolled steel, 5-V, as compared with the stabilized steel, 5-IX, suggests that the composition of the ferrite may be re-

sponsible, the faster cool retaining more elements in solution thereby giving rise to the steps.

0.26 Per Cent Carbon Steel: Like the 0.10 per cent carbon steel the as-rolled and normalized 0.26 per cent carbon steels looked alike under the microscope (Fig. 18), while the pearlitic areas of the stabilized steel are somewhat more rounded (Fig. 19). The break-up of the pearlite by the stabilizing treatment is clear. It will be observed also that the cementite fills in much of the space between the ferrite crystals. There was a decrease of 8 points in Rockwell B due to the stabilizing.

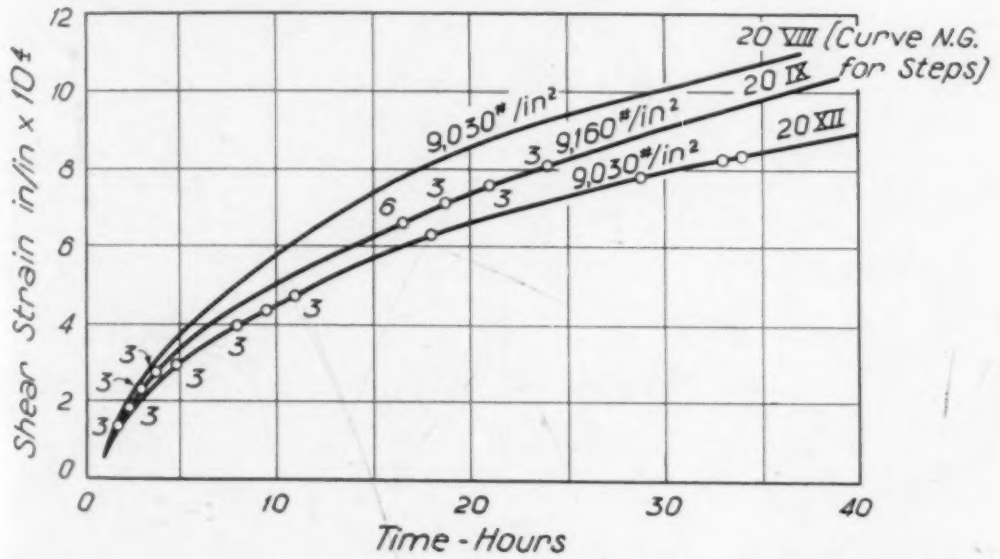


Fig. 15—Creep Curves of Stabilized 0.26 Per Cent Carbon Steel at 350 Degrees Cent. (660 Degrees Fahr.).

A striking example of the effect of stabilizing the 0.26 per cent carbon steel on decreasing the resistance is shown by curves 10-V, as-rolled, and 10-II, stabilized (Fig. 12). Both were run under nearly identical conditions but the creep rate of the as-rolled steel during the fifth to tenth hours was 35×10^{-4} per cent per hour while that of the stabilized was 310×10^{-4} per cent per hour.

It will be recalled that steels 10 and 20 were purchased separately and stabilized separately but the compositions were the same. Stabilized steel 20 was, however, about 4 points harder (68-B) than 10 and this is reflected in the creep curves of the two (Fig. 12). Curve 20-VI shows steel 20 and 10-II steel 10 under the same stress; the former being somewhat stronger.

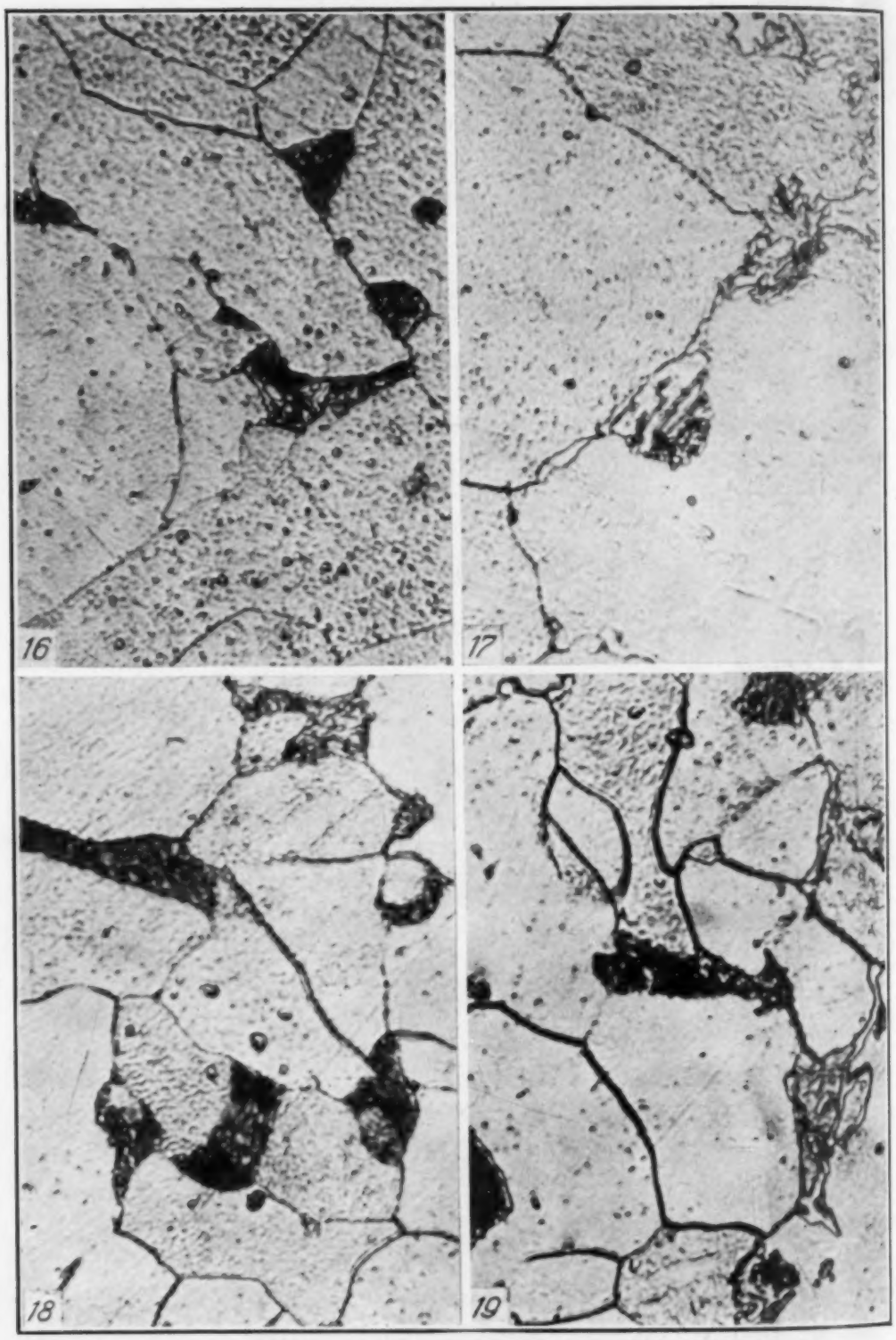


Fig. 16—Photomicrograph of 0.10 Per Cent Carbon Steel in the Normalized Condition.
Fig. 17—Same Steel After Stabilizing.
Fig. 18—Photomicrograph of 0.26 Per Cent Carbon Steel in the Normalized Condition.
Fig. 19—Same Steel After Stabilizing.

It might be pointed out that R. W. Bailey found a decrease of 25 per cent in tensile creep strength of a fully spheroidized eutectoid steel as compared with the normalized steel.

In no case was there observed any selective grain growth which might arise from the strain gradient existing in a strained solid torsional specimen. Most likely the maximum temperature of 500 degrees Cent. (930 degrees Fahr.) used was too low to give rise to this phenomenon, but P. Henry (1) found it to occur at 650 degrees Cent. (1200 degrees Fahr.).

Table II
Physical Data on Steels Compared

	Designation	Per Cent C	Mn	Si	P	S	A.S.T.M.* Grain Size (Ferrite)	Tensile Strength Rm. Temp.	Per Cent RA
Tested in Torsional Creep Present Tests	10AR	0.25	0.54	0.04	0.007	0.025	7	66,000	64
	10N	0.25	0.54	0.04	0.007	0.025	7		
	10S	0.25	0.54	0.04	0.007	0.025	7		
	20S	0.26	0.54	0.03	0.006	0.033	7	60,100	61
	5AR	0.10	0.50	0.02	0.025	0.065	6	55,500	66
	5N	0.10	0.50	0.02	0.025	0.065	6	54,000	68
	5S	0.10	0.50	0.02	0.025	0.065	6	51,000	71
Tested by Pomp and Höger in Tensile Creep	AI	0.11	0.36	trace	0.009	0.018	6	52,200	61
	BI	0.09	0.42	trace	0.020	0.026	4	50,200	60
	CI	0.08	0.45	trace	0.012	0.022	6	49,600	63
	D	0.10	0.39	trace	0.052	0.056	–	52,700	72
	E	0.10	0.55	0.06	0.027	0.030	–	57,200	76
	AIV	0.22	0.61	0.10	0.015	0.028	7	66,100	53
	C3**	0.26	0.78	0.27	0.039	0.043	6–7	76,000	55

*All grain sizes were determined by the author from the published photomicrographs.
**From Pomp and Enders.

The Relation of Creep in Torsion to Creep in Tension

Immediately after applying the test load to a torsional creep specimen the stress distribution in one extreme, assuming for the moment no immediate plastic flow, is as shown in Fig. 20a. Here the circle represents the cross section of the specimen, the abscissa the radius, and the ordinate the stress. The stresses for this elastic condition are zero at the axis and increase linearly to a maximum at the surface. The surface stress is given by the equation,

$$f_m = \frac{16T}{\pi d^3} \quad \ldots\ldots\ldots \quad (1)$$

where

f_m = surface stress
T = applied torque
d = diameter of specimen

With the advent of plastic flow, however, the stress distribution may in the extreme alter itself so as to be represented by Fig. 20b if the deformation is sufficient and the metal does not work harden in the final stages of deformation. Fig. 20b shows the stresses to be the same at all radii; the stress is given by the equation,

$$f' = \frac{12T}{\pi d^3} \quad \ldots\ldots\ldots \quad (2).$$

It will be observed that it is possible for the surface stress to decrease to 0.75 of its initial value by this change.

In actuality, however, it is not likely that the stress will become entirely uniform during the process of creep, but will assume an arbitrary distribution as represented in Fig. 20c. The change in stresses at the various radii are represented by Fig. 20d; the stresses in the outer radii decrease and the inner radii increase. Under the arbitrary conditions the peripheral stress is somewhere between f_m and 0.75 f_m. It is desirable to determine more precisely what this arbitrary stress is. In so doing we may consider the following analogy.

Referring to Fig. 21 let us assume that two tensile specimens S_1 and S_2 are suspended on pins so that they are free to pivot. Let stresses p_1 and p_2 be applied to both bars by means of the force P on a bar pivoting about O; then for the dimensions shown, if the bar is perfectly rigid, S_2 will elongate two times as much as S_1, and if elastic strains only occur, the stress on S_2 will be twice that on S_1*. But experiments have shown that S_2 wants to creep more than twice as fast as S_1. Such being the case specimen S_2 after a time, if it were not tied with S_1, would creep to a point a_2 and specimen S_1 would creep to a point a_1, but inasmuch as they must creep together they creep to arbitrary points x_2 and x_1, where $x_1 = \frac{x_2}{2}$. But specimen S_1 has moved farther than it wanted to and additional stress must of necessity be imposed on it to account for this difference, and, because the torque remains the same, the stress on S_2 must decrease. This automatic adjustment of stresses has to go on until the stress in specimen S_1 is sufficient to cause it to creep one-half as fast as specimen S_2. For example if we assume that creep rate Ⓓ varies

*Even though plastic strain occurred here the results of this analysis would not be changed.

as f^3 then P_2 which was originally related to stress p_1 by $p_2 = 2p_1$ becomes,

$$p_2 = \sqrt[3]{2}\, p_1 = 1.26\, p_1$$

so that the stress on each bar comes closer to the other. Only in the case of viscous flow, where stress and flow are linear, would the stresses on both specimens remain the same.

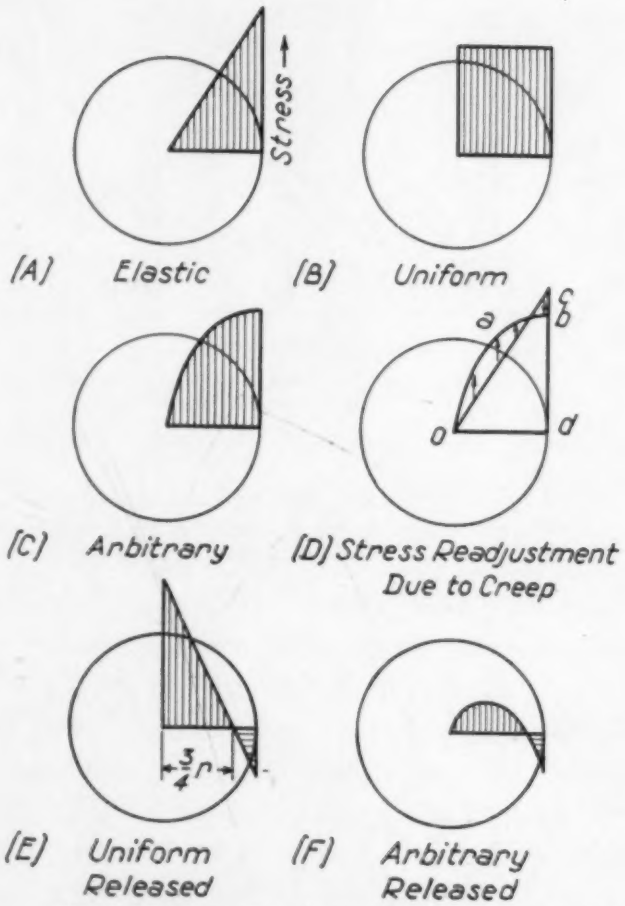


Fig. 20—Stress Distribution in Solid Cylindrical Torsion Specimens.

The stress conditions in a solid cylindrical bar creeping in torsion may be considered analogous to the example just given. That is, with each radius creeping at a rate directly proportional to that radius, the stress at each radius (or concentric cylinder of thickness dt) is increased or diminished so that at equilibrium the stress at each radius is just sufficient to produce this creep rate. Of course each assumed concentric cylinder of the test specimen is not rigidly fixed to its neighbor as in the analogy given but instead each cylinder

might possibly creep with respect to its neighbor. This, however, would merely mean that the time required for equilibrium would be increased somewhat.

Now by plotting creep rates ranging from zero at the center linearly to a maximum at the periphery as abscissas; and by plotting the stresses necessary to produce these creep rates as ordinates it is

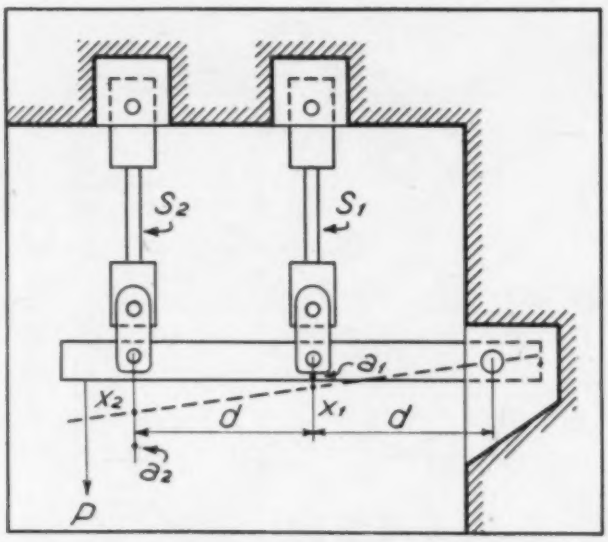


Fig. 21—Diagrammatic Sketch Showing Assembly of Two Tensile Test Specimens so Arranged to Pivot Freely.

possible to determine the shape of the arbitrary curve. From the equation of this curve and knowing the torque applied it is possible to determine the peripheral or maximum stress. For data we may resort to the results of tensile tests. Pomp and his co-workers have published many data which may be applied. Applying their data for steels containing about 0.10 per cent carbon at 400 degrees Cent. (750 degrees Fahr.) various curves such as those shown in Fig. 22 were obtained. In general the curves more nearly approach a linear relationship the lower the peripheral creep rate assumed. In order to determine how much these curves would vary assuming the same peripheral creep rate for each steel, all the curves equivalent to a rate of 50×10^{-4} per cent per hour were plotted on one graph; the spread of the resulting curves is shown in Fig. 23. There is a considerable spread among them as will be observed.

Bailey has already expressed the peripheral stress resulting from logarithmic and semi-logarithmic curves in terms of the stress resulting with uniform distribution.

In other words if creep rates are related to stress by either of the two following general equations (3 or 4) the peripheral stress resulting can be determined:

$$f = a + n\ln\textcircled{D} \quad \ldots\ldots\ldots \quad (3)$$
$$\log_{10} f = a' + b \log_{10}\textcircled{D} \quad \ldots\ldots \quad (4)$$

where

f = stress
n, a, a' and b = constants
$\ln\textcircled{D}$ = logarithm of creep rate to the base e.
$\log_{10}\textcircled{D}$ = logarithm of creep rate to base 10.

The peripheral stress is given in terms of uniform stress by the relationship

$$f = f' + \frac{n}{3} \text{ and}$$

$$f = (1 + \frac{b}{3}) f'$$

where f′ equals stress through the cross section if the stress were uniform. f′ can easily enough be calculated from the equation (2). In other words with a semi-logarithmic creep curve the peripheral stress is $\frac{n}{3}$ greater than that resulting when assuming uniform stress, and with a logarithmic curve it is $\frac{b}{3}$ f′ greater.

The equations for the high and low curves of Fig. 23 were determined. The high curve followed the logarithmic equation:

$$\log f = 0.15 \log \textcircled{D} + 1.7 \quad \ldots\ldots\ldots \quad (4a)$$

This is plotted in Fig. 23. Here b = 0.15 and $\frac{b}{3} = 0.05$ so that

$$f = f'(1 + 0.05) = 1.05f'$$

or the peripheral stress is 5 per cent greater than the easily calculable uniform stress.

The low curve followed closely the semi-logarithmic equation:

$$f = 27.3 \ln\textcircled{D} - 26 \quad \ldots\ldots\ldots \quad (3a)$$

This also is plotted in Fig. 23. Here $n = 27.3$ or $\frac{n}{3} = 9.1$ so that

$$f = f' + 9.1 = 100 \text{ or}$$
$$f = 1.10f'.$$

In other words the peripheral stress is 10 per cent greater than that assuming uniform stress.

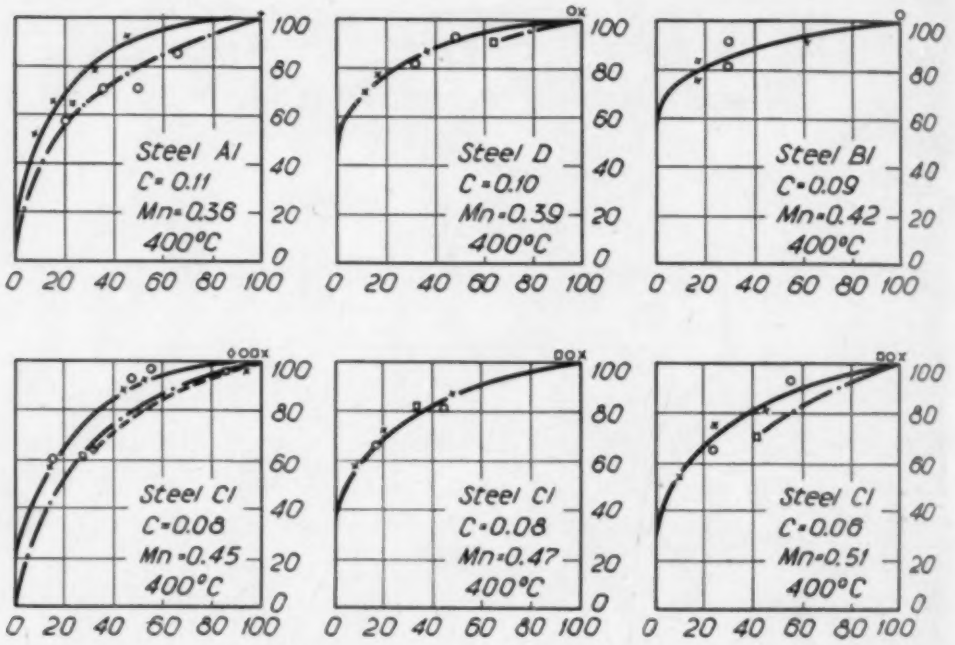


Fig. 22—Probable Stress Distribution in Solid, Cylindrical Torsional Creep Specimens at Equilibrium as Deduced from Tensile Data for Various Low Carbon Steels and Creep Rates. (Note: The abscissae are "Relative Creep Rate," the ordinates "Relative Stress."

It is seen then that even with the widely separated curves shown in Fig. 23, the peripheral stress only varies 5 per cent in terms of the uniform condition. As a mean then, $f = 1.07\ f'$.

Upon plotting the data of Pomp and his co-workers for steels up to 0.25 per cent carbon and at 500 degrees Cent. (930 degrees Fahr.) as well as 400 degrees Cent. (750 degrees Fahr.) it was found that the great majority of the resulting curves fell between the limits shown in Fig. 23, represented by equations 3a and 4a, so that 107 per cent f′ may be taken as typical.

Bailey, as quoted previously, was of the opinion that the stress could be considered as uniform throughout the cross section.

Having now arrived at a value of

$$f = 1.07 \frac{12T}{\pi d^3}$$

as the maximum (peripheral) shear stress in a solid cylindrical specimen after sufficient creep has occurred to produce equilibrium stress conditions, it is possible to determine the relationship between the stresses and creep rates as found in the simple tensile test and pure shear. In making these comparisons it is necessary to convert tensile stresses to equivalent shear stresses and tensile creep rates to equivalent shear creep rates. For a simple tensile stress t the equivalent shear stress f = ½ t. G. H. MacCullough (11) has shown that a tensile creep rate c is equivalent to a shear creep rate Φ = 2c. The latter relationship means that the shear creep rate Φ in straight tension occurring at 45 degrees to the axis of pull is equal to twice the tensile creep rate c in the direction of pull. Likewise, the shear creep rate Φ in pure shear taking place at 90 degrees to the axis of the specimen is equal to twice the tensile creep rate c occurring at 45 degrees to the axis. Thus in the present investigation when comparing the tensile data of Pomp and his co-workers with the present torsional data it was necessary to halve their stresses and double their creep rates.

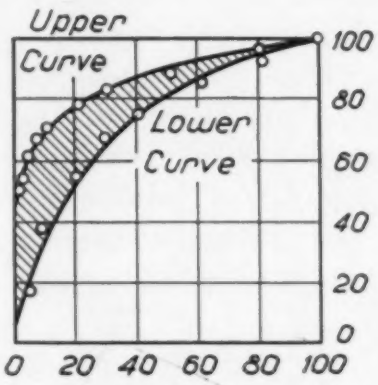


Fig. 23—Spread of Curves Representing Peripheral Creep of 50×10^{-4} Per Cent Per Hour. Abscissa = Relative Creep Rate; Ordinate = Relative Stress.

In the above discussion the results (namely 1.07f′) depended upon equilibrium (or nearly so), and the question arises as to how long a time or how much creep is necessary before equilibrium is attained. It appears that this is a very difficult problem and one which might well invite criticism of the method. There appears,

however, to be two methods permitting an approximation of this value.

First, if we assume for the moment that if the stress distribution in torsional creep remained the same with time, we would expect a time-deformation curve similar to a tensile time-deformation curve; that is the ratio of the creep rate during the third to sixth hours to that in the fifth to tenth hours would be the same in each case. It has been stated, however, that in torsional creep the peripheral stress decreases with time which would mean that the above mentioned ratio would be higher in torsion than in tension unless of course equilibrium had been practically reached prior to the third to sixth hour interval. Unfortunately the results of the present tests do not permit of making such comparisons. Pomp and Höger give numerous values for the ratios of the creep rates in the third to sixth and twenty-fifth to thirty-fifth hours to those in the fifth to tenth hours but the ratios differ appreciably for each steel. To arrive at any satisfactory results it would be necessary for tensile and torsional creep rates to be determined on the same steels.

Second, in accordance with Fig. 20d it should be possible to choose any radius and determine how much twist is necessary to increase the stress from the elastic to the arbitrary curve. For this determination it is assumed that the stresses build up elastically at the inner radius chosen as the outer radii creep. This assumption means that the value of twist obtained will necessarily be a *minimum* value because some plastic creep will simultaneously be taking place even at the inner radius chosen.

Comparison of Results with Those of Pomp and Co-workers

The results of the comparisons are given in Table III. The last column of this table gives the relationships existing between the equivalent shear stress of simple tension and simple shear stress for the same shear creep rates; the average is 1.20. From this value it is possible to calculate what the shear creep rates would be if the equivalent shear stress in tension is made equal to the shear stress in torsion. Equation (4) may be rewritten into the form:

$$\Phi = Af^{1/b} = Af^{B} \ldots\ldots\ldots \quad (4b)$$

where A is a constant and $B = \frac{1}{b} = \frac{1}{.15} = 6.67$. This value checks

closely with that given by Bailey according to whom a value of 6 may be taken for carbon steels and probably also for low alloy steels. Thus in this stress range the creep rate varies as the sixth power of the stress. Therefore $1.2^6 = 3.0$ is the factor by which the equivalent shear creep rates in tension should be multiplied to obtain values comparable to shear creep rates in torsion. In other words $\Phi_t = 3\Phi_s$ or $c = 1.5\Phi_s$;

where Φ_t = shear creep rate in tension
Φ_s = shear creep rate in torsion
and c = tensile creep rate in tension.

What significance can be attached to the relationship $\Phi_t = 3\Phi_s$? Bailey has already shown with tests on 0.115 per cent carbon* steel tubes that at 480 degrees Cent. (900 degrees Fahr.)

$$\Phi_t = 1.9\Phi_s \text{ to } 2.3\Phi_s$$

and at 550 degrees Cent. (1020 degrees Fahr.)

$$\Phi_t = 2.7\Phi_s \text{ to } 3.2\Phi_s.$$

Table III
Relation of Tensile to Torsional Creep Stresses for Certain Creep Rates During Fifth-Tenth Hours

Steels Compared	Shear Creep Rate	Tensile Stress (Equivalent Shear)	Torsional Stress	Ratio of Stresses
Tests at 400 Degrees Cent. (750 Degrees Fahr.)				
5AR	12	4,800	6,350	1.32
vs.	26	6,400	8,190	1.28
AI	52	7,800	9,410	1.21
5N vs. AI	25	6,450	8,190	1,27
5AR vs.	26	7,100	8,190	1.15
BI	52	7,700	9,410	1.22
5N vs. BI	107	8,800	10,800	1.23
5AR vs.	26	7,800	8,190	1.05
CI	52	8,200	9,410	1.15
5N vs. CI	107	8,950	10,800	1.21
5AR vs. D	26	7,400	8,190	1.11
	52	8,300	9,410	1.14
5N vs. D	107	8,800	10,800	1.23
10AR vs. C3	35	9,800	10,280	1.05
Tests at 300 Degrees Cent. (570 Degrees Fahr.)				
5AR vs. AI and BI	18	15,400	18,400	1.19
			Average	1.2

*Previously annealed and held 1 hour at 650 degrees Cent. (1200 degrees Fahr.).

If the maximum shear stress alone were the criterion of failure it would be expected that $\Phi_t = \Phi_s$ inasmuch as the maximum shear stresses are the same in each case. Bailey, however, had anticipated a value of $\Phi_t = 2\Phi_s$. The reason for this may be seen from Fig. 24 in which Fig. 24A represents the principal stresses acting in tension and Fig. 24B those in torsion; the maximum shear stress f in each case being equal to $\frac{Y}{2}$. Bailey states first, that creep is general and not confined to planes of maximum shear stress and second, that the shear stress on all planes parallel to the Z axis is the same in both

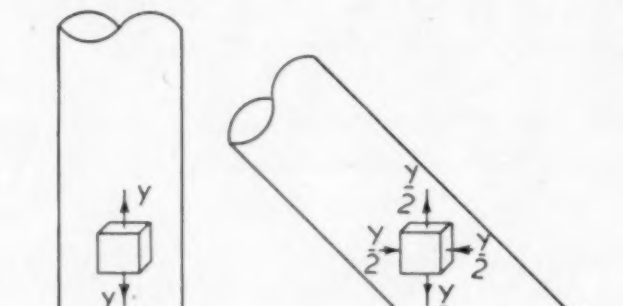


Fig. 24—(a) Diagrammatic Sketch Illustrating Principal Stresses in Tension and (b) Those in Torsion.

cases; but in Fig. 24A the shear stress on all planes parallel to the X axis is similar to that on corresponding planes parallel to the Z axis while in Fig. 24B the shear stress on planes parallel to the X axis is one-half that on the corresponding planes in Fig. 24a and less creep in the direction of the stress Y would be expected. This would mean that the creep in Fig. 24A should not exceed twice that of Fig. 24B. However, as Bailey found $\Theta_t > 2\,\Phi_s$ it was suggested that "it is reasonable to believe that creep due to shear on a set of planes parallel to one coördinate axis is capable of being influenced by shear stresses on the three sets of planes parallel to all three coördinate axes; in other words, by the general system of shear stress existing."

Is it not possible to explain the relationship $\Phi_t > 2\Phi_s$ as a modification of Bailey's view as follows: In torsion there is only one

plane of maximum shear stress contributing to measurable creep; namely, normal to the axis of twist. In tension, however, there are an infinite number of planes on which creep may take place, that is, on all planes making an angle of 45 degrees with the direction of pull. In tension, therefore, creep may choose its own plane or shift from one plane to another according to which is weaker at the moment—creep on one plane will strengthen that plane and further creep will occur on another plane. In torsion there is no alternative thus making creep a slower process. The same reasoning would hold true if creep were general.

Acknowledgments

The author wishes to especially thank Professor Albert Sauveur of Harvard University under whom this work was carried out and who suggested the problem. Professor Sauveur is an ardent advocate of torsional testing and he is largely responsible for the development of the machine described. Thanks are also due to: Prof. F. H. Norton and Dr. J. A. Fellows of the Massachusetts Institute of Technology for building the furnace and offering valuable suggestions, and to Dr. G. H. MacCullough for reading the paper and making valuable suggestions.

Bibliography

1. P. Henry, "Recherches Experimentales Sur Les Vitesses de Deformation des Metaux aux Hautes Temperatures," *Revue de Metallurgie,* Vol. 24, No. 1, 1927, p. 421-42.
2. R. W. Bailey, *Transactions,* Tokyo Sectional Meeting, World Power Conference, Tokyo, Vol. 3, 1929, p. 1089-1121. See also "Creep of Steel Under Simple and Compound Stresses," *Engineering,* Vol. 129, 1930, p. 265-66 and 327-29. See also "Thick Walled Tubes and Cylinders Under High Pressure and Temperature," Ibid, p. 772-73, 785-86 and 818-19.
3. F. L. Everett, "Strength of Materials Subjected to Shear at High Temperature," American Society of Mechanical Engineers, Vol. 53, 1931, p. 117-129. See also "Creep of Metals in Shear at High Temperatures" *Physics,* Vol. 4, 1933, p. 119-121.
4. G. R. Brophy, "The Characteristics of Deformation of Steel Under Constant Load at Elevated Temperatures," American Society for Steel Treating, Vol. XX, No. 1, 1932, p. 58-66. See also "Time Deformation —the Nature of Creep Tests," Metal Progress, Vol. XX, No. 2, 1931, p. 54-57 and 96.
5. F. C. Lea and C. F. Parker, "The Effect of Temperature on Some of the Physical Properties of Metals," *Engineering,* Vol. 133, 1932, p. 23-26 and 54-55.

6. F. H. Norton and J. A. Fellows, "A New Device for Creep Testing," METAL PROGRESS, Vol. XXIV, No. 4, 1933, p. 41-43.
7. A. Pomp and W. Höger, "Dauerstandfestigkeitsuntersuchungen an Kohlenstoff und niedriglegierten Stählen nach dem Abkurzungsverfahren," *Mitt. aus dem Kaiser-Wilhelm Inst. für Eisenforsch.,* Düsseldorf, Vol. XIV, 1932, p. 37-57.
8. A. Pomp and A. Dahmen, "Entwicklung eines abgekürtzten Prüfverfahrens zur Ermittlung der Dauerstandfestigkeit von Stahl bei erhöhten Temperaturen," *Mitt. aus dem Kaiser-Wilhelm Inst. für Eisenforsch.,* Düsseldorf, Vol. IX, No. 3, 1927, p. 38.
9. A. Pomp and W. Enders, *Mitt. aus dem Kaiser-Wilhelm Inst. für Eisenforsch.,* Düsseldorf, Vol. XII, 1930, p. 127. See also "Abkürzungsverfahren zur Bestimmung der Dauerstandfestigkeit des Stahles," *Inst. Assoc. for Test. Mat.,* Zurich, Vol. I, 1932, p. 143-159. See also "Accelerated Method for Determining Creep Limit," *Instruments,* Vol. 5, No. 7, 1932, p. 166-168.
10. F. Körber and A. Pomp, "Festigkeitseigenschaften von Kesselblechen bei erhöhten Temperaturen mit besonderen Berucksichtigung des Verhaltnisses der Warmtreckgrenze zur Zugfestigkeit bei Raum-temperatur, *Mitt. aus dem Kaiser-Wilhelm Inst. für Eisenforsch.,* Düsseldorf, Vol. XII, 1930, p. 12-32.
11. G. H. MacCullough, "Applications of Creep Tests," *Transactions,* American Society of Mechanical Engineers, Applied Mechanics Division, Vol. I, No. 3, 1933, p. 87-97.
12. R. W. Bailey, "The Utilization of Creep Test Data in Engineering Design," Institute of Mechanical Engineer, Adv. Copy, Nov. 1935, p. 136.

A STUDY OF THE A_0 POINT OF IRON BY DIFFUSION OF HYDROGEN

By W. R. Ham and W. L. Rast

Abstract

By diffusion of hydrogen through the metal a careful study has been made of the thermal critical points occurring between 55 and 920 degrees Cent. in samples of iron practically pure except for small determined amounts of carbon. Log_{10} Rate vs. $\frac{1}{T^0K}$ isobars for five samples exhibit characteristic discontinuities accompanied by hysteresis at the crystal structure transformation (A_3), at the magnetic transition (A_2), and in the A_0 region. Controlled decarburization was effected by heat treating carbonyl iron samples for known times in hydrogen at 1100 to 1200 degrees Cent. (2010 to 2190 degrees Fahr.). The amount of carbon remaining in solution after annealing was estimated from the slope of the linear isotherm log_{10} Rate vs. log_{10} Pressure graph, taken at 500 degrees Cent. (930 degrees Fahr.) (theoretical slope for pure iron is one-half). Critical temperatures are recorded for samples having 500 degrees Cent. (930 degrees Fahr.) isothermal slopes of 0.555, 0.550, 0.535, and 0.520. The last sample has critical points as follows: Ar_0 at 315 to 303 degrees Cent.; Ac_2 at 729 to 767 degrees Cent.; Ac_3 at 902 to 908 degrees Cent. The slopes of the linear isotherms show anomalous behavior in the A_0 region. The thermal position of the A_0 discontinuity is very sensitive to change in carbon content; but apparently the discontinuity cannot be removed by complete decarburization, and is believed to be due to electronic rearrangement in the iron lattice.

THIS study is an extension of work previously published in this journal by one of the authors (1)[1]. The main objective of the entire investigation has been to make a detailed study of the thermal critical points of nearly pure iron by the diffusion of hydrogen method.

[1]The figures appearing in parentheses refer to the bibliography appended to this paper.

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Many investigators have studied the diffusion of gases through metals as a function of sample temperature and gas pressure. The diffusion of hydrogen through iron has previously been studied in some detail by Borelius and Lindblom (2), by Smithells and Ransley (3), and by one of the present authors (loc. cit.). Of these three investigations only the latter involves a sufficiently large number of experimental observations taken over a range of temperature wide enough to render the results of value in a study of the thermal critical points of the iron. Furthermore, both of the former investigations were performed upon samples apparently containing small but important amounts of carbon in solution in the iron.

In the present work the authors have devoted their efforts to a careful study of the thermal critical points occurring between 55 and 920 degrees Cent. (130 to 1690 degrees Fahr.) in samples of iron practically pure except for small controlled amounts of carbon. Especial attention has been given to the so-called A_0 point previously noted in iron-carbon alloys and ascribed by some to the magnetic transformation of cementite (Fe_3C). Of particular interest is the fact that a pronounced "discontinuity" occurs in the rate of diffusion versus temperature curves for all samples of iron investigated by the authors at temperatures comparable with those at which the A_0 point is known to occur. Moreover, this diffusion discontinuity apparently cannot be removed by complete decarburization. At least it is still present in the No. 1 hydrogen purified iron of Mehl (4). The effect of carbon contamination upon the A_2 and A_3 critical points of iron has been examined by the authors and is found to be in agreement with generally accepted views.

A consideration of the nature of the diffusion process makes it appear reasonable that any change inside the metal which affects the equilibrium spacing of the lattice points, their force interactions, or their motions should produce a pronounced effect upon the diffusing gas ions or excited atoms.

Experimental Technique

Two separate sets of diffusion apparatus, of the general type previously described by Ham (loc. cit.), were used during the entire course of the experiment. Two different methods of observation of the rate of diffusion were employed interchangeably: namely, the "trapped" method and the "constant flow" method. These methods

previously have been discussed elsewhere, but a short description here seems worthwhile. In the "trapped" method the gas diffuses through a sample at a constant temperature into a previously evacuated fixed volume and the diffusion rate is taken as proportional to the time rate of increase in pressure as measured with a McLeod gage and stop-watch. If the volume in which the diffusing gas collects is known, the absolute rate of diffusion measured in grams of gas diffusing through a square centimeter of sample surface per second for one millimeter thickness of the sample can be computed from the rate as observed in microns of mercury per minute. Here the absolute rate of diffusion is computed for only one sample, and these results are approximate, since the area of the sample was not determined with care. In the "constant flow" method the gas again diffuses into a fixed volume but is permitted to escape continuously through a tiny aperture (stop-cock) into a highly evacuated space. The absolute level of the equilibrium pressure in the fixed volume is found both theoretically and experimentally to be directly proportional to the rate of diffusion; thus McLeod gage readings can be used as a measure of the diffusion rate. Use of the "constant flow" method minimizes errors due to slow absorption or evolution of gas by the glass and metal parts of the apparatus. For the measurement of low diffusion rates, where the time of trapping is long, considerable error can be introduced by this absorption or evolution. The authors frequently multiplied the McLeod gage readings in the "constant flow" method by a factor as great as thirty by decreasing sufficiently the size of the small aperture.

The method of mounting the iron samples is shown diagrammatically in Figs. 1, 2 and 3. A small disk of the "sample" iron of thickness about 0.1 inch was clamped between two short pieces of Armco iron tubing, as shown in Fig. 1, and atomic hydrogen welded, while constant streams of hydrogen washed the sample faces. The sample and the Armco mount were then machined down, the thickness of the "sample" disk being reduced to 0.03—0.06 inches. Decarburization was effected by placing the machined piece inside a cylindrical nickel bomb lined with sheet iron and heat treating in an hydrogen atmosphere at 1100 degrees Cent. (2010 degrees Fahr.) for as many hours as desired. The hydrogen flowed in a steady stream directly from a commercial tank through an iron tube into the bottom of the bomb and burned as it escaped through a small opening at the top. Next, following the method of Ham (loc. cit.), the decarburized

piece was copper welded into a nickel sleeve, as shown in Fig. 2. Finally, sections of nickel tubing were copper welded into the open ends of the nickel sleeve, as shown in Fig. 3. The sample was carefully protected during the welding process by removable iron baffle plates, and all heating was done in an atmosphere of hydrogen.

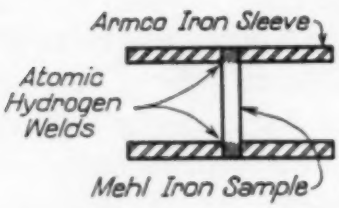

Fig. 1—First Stage of Diffuser Assembly, Showing Atomic Hydrogen Weld of Iron Sample to Armco Iron Sleeve.

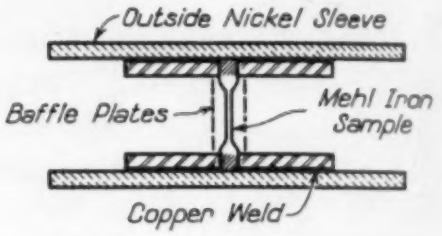

Fig. 2—Second Stage of Diffuser Assembly, Showing Copper Weld.

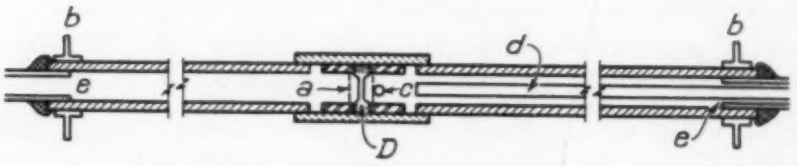

Fig. 3—Final Diffuser Assembly.

The samples prepared as indicated were free from any appreciable metallic contamination and remained so in the diffusion apparatus unless they were heat treated for several hours at or above 1000 degrees Cent. (1830 degrees Fahr.). Prolonged heat treating at very high temperatures probably resulted in the deposition of nickel vapor upon the sample faces. The authors were very careful to avoid excessive heating of the samples during the process of making observations, the diffusion rates below 500 degrees Cent. (930 degrees Fahr.), rather than those above, being measured first. However, in all cases observations made subsequent to short studies of the diffusion rates at temperatures as high as 920 degrees Cent. (1690 degrees Fahr.) indicated no detectable contamination.

The commercial hydrogen used in the experiment was freed of water vapor and oxygen by passing it through calcium chloride and phosphorous pentoxide drying tubes and through a heated tube filled with pumice stone covered with chemically deposited nickel. How-

ever, removal of these purification tubes seemed to have no measurable effect on the results obtained.

After changing samples the observers recorded no data until the vacuum systems were properly degassed and vacuua of the order of 0.001 micron were obtained.

The McLeod gages were carefully calibrated when built and were in addition checked for irregularities of capillary bore by trapping of the hydrogen when it was diffusing at a constant rate.

Temperatures were measured by chromel-alumel thermocouples

Table I

Sample No.	Thickness	Type	Treatment
1	48 mils	"Yensen"	
2	63	Carbonyl	Not heat treated.
3	61	Carbonyl	Annealed 5 hours at 1100 degrees Cent. in H_2.
4	54	Carbonyl	Annealed 8 hours at 1100 degrees Cent. in H_2.
5	32	"Mehl"	Annealed 250 hours at 1200 degrees Cent. in H_2.

calibrated in site, for each iron sample, against the melting point of 99.992 per cent silver obtained from the U. S. Bureau of Standards. Thermostat circuits of the photocell control type held the sample temperatures constant within about 1 degree Cent.

Care was exercised to obtain equilibrium of the diffusion rate in every case. For work at atmospheric pressure and temperatures above 700 degrees Cent. (1290 degrees Fahr.) this presented no great difficulty, but at impressed pressures of 10 centimeters of mercury or lower and at temperatures below 300 degrees Cent. (570 degrees Fahr.) eight or ten hours were often necessary.

Experimental Results

Measurements were made on five samples of iron, the types and treatments of which are listed in Table I. The iron for Sample No. 1 was obtained from the Westinghouse Company (kindly supplied by Dr. T. D. Yensen) and is referred to as Yensen iron. Samples No. 2, 3 and 4 were carbonyl iron of German origin, nearly free from all impurities save carbon. Sample No. 2 was not decarburized, while Samples No. 3 and 4 were partially freed of carbon by treating in hydrogen at 1100 degrees Cent. (2010 degrees Fahr.) for times of five and eight hours respectively. The high purity carbonyl iron for Sample No. 5 was obtained from the Metals Research Laboratory of the Carnegie Institute of Technology (kindly supplied by Dr. R. F.

Mehl). Wells, Ackley, and Mehl (loc. cit.) carefully purified this iron by heat treating for 250 hours at 1200 degrees Cent. (2190 degrees Fahr.) in hydrogen.

Curve D of Fig. 4 is the diffusion isobar or $\log_{10}$ Rate vs. $1/T°K$ graph at atmospheric pressure obtained for the sample of

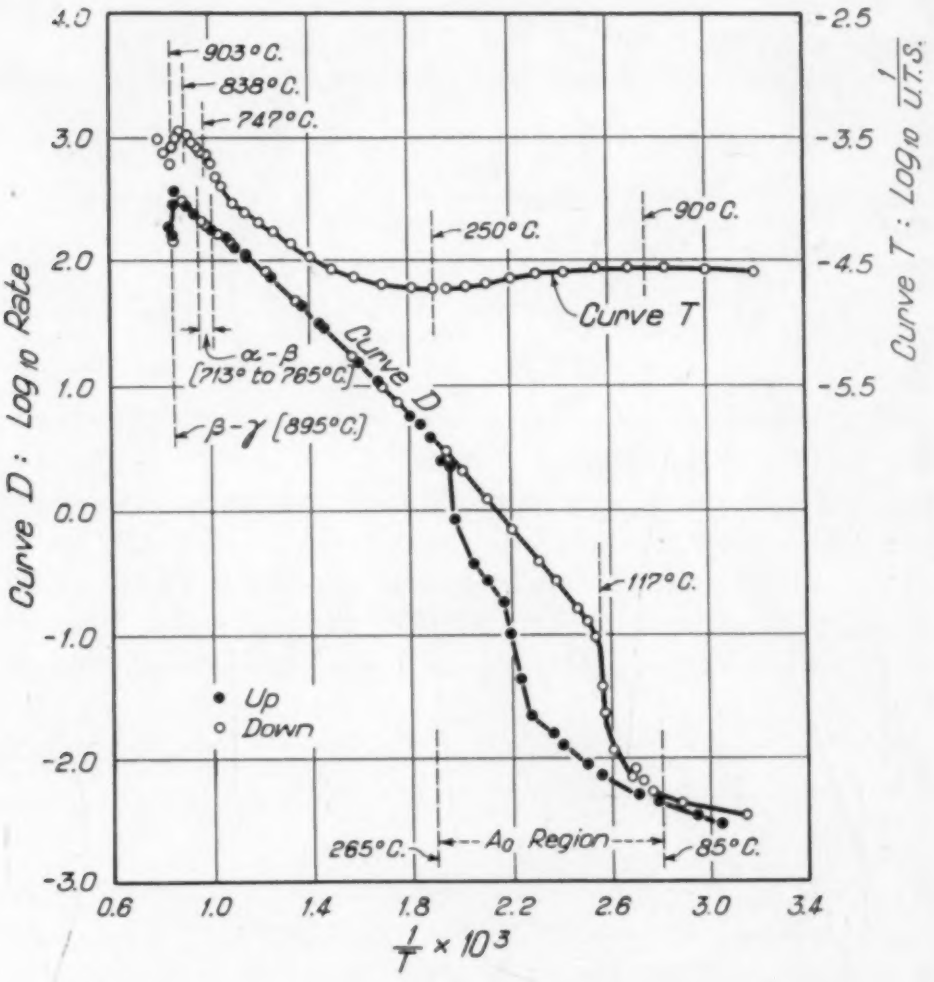

Fig. 4—Curve D: Diffusion Isobar (55-920 Degrees Cent.) at Atmospheric Pressure for Nondecarburized Carbonyl Iron—"Constant Flow" Method. Curve T: Ultimate Tensile Strength Plot for Electrolytic Iron.

nondecarburized carbonyl iron in the temperature range 55 to 920 degrees Cent. (130 to 1690 degrees Fahr.). The isobars for all samples show "discontinuities," accompanied by hysteresis, in the low temperature range, in the vicinity of the A_2 or magnetic transition, and in the vicinity of the A_3 or crystal structure transformation.

The diffusion rate appears to obey the formula

$$R = AP^{y}T^{z}\, e^{\frac{-b}{T}}, \qquad (1)$$

where R is the diffusion rate
A is a constant
P is the gas pressure on the ingoing side of the sample, a vacuum (approx.) existing on the outgoing side
T is the absolute temperature
e is the natural logarithmic base
b is the net diffusion work function in degrees Kelvin for the gas metal system
y takes on values between 0.5 and 1.00 (approx.) depending upon the state of purity of the sample and the value of the temperature
z is a constant at present unknown with precision but probably having a negative value of the order of unity.

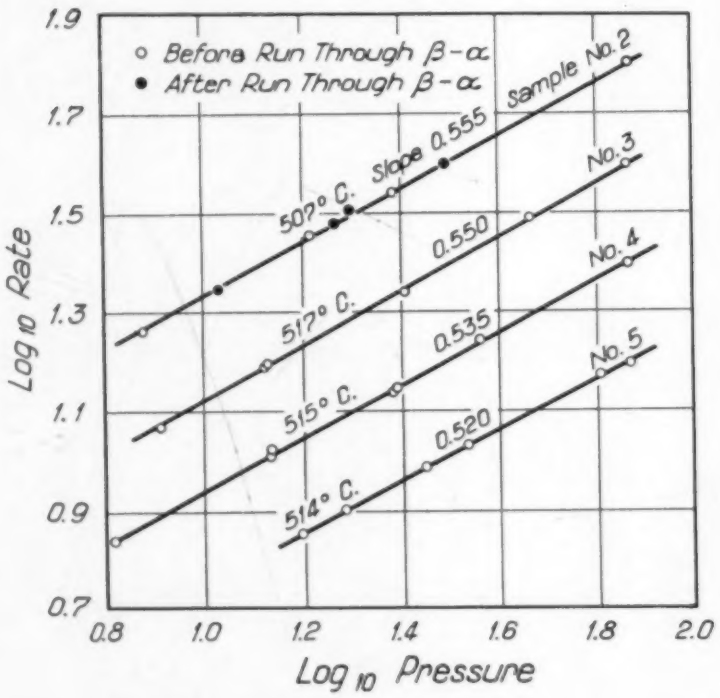

Fig. 5—Diffusion Isothermals at 500 Degrees Cent. (Approx.) for Samples No. 2-5 Inclusive.

In every instance a slight but definite concave curvature toward the axis of abscissae was found in the $\log_{10}$ Rate vs. $1/T^{\circ}K$ isobars between the A_2 critical region and the low temperature discontinuity. This indicates that z is a small negative number, but an accurate determination of its value is rendered difficult by the increase of y with increase in carbon content or with decrease of temperature.

Since the curvature in the diffusion isobars is considerable for

temperatures between about 550 and 730 degrees Cent. (130 to 1345 degrees Fahr.), the "slopes" of the isobars (values of "b" in Equation I) are recorded for a temperature range of 300 to 550 degrees Cent. (570 to 1020 degrees Fahr.), in which the isobars are more nearly linear. Where the precision of the data permits, the "slopes" of the isobars at very low temperatures are given.

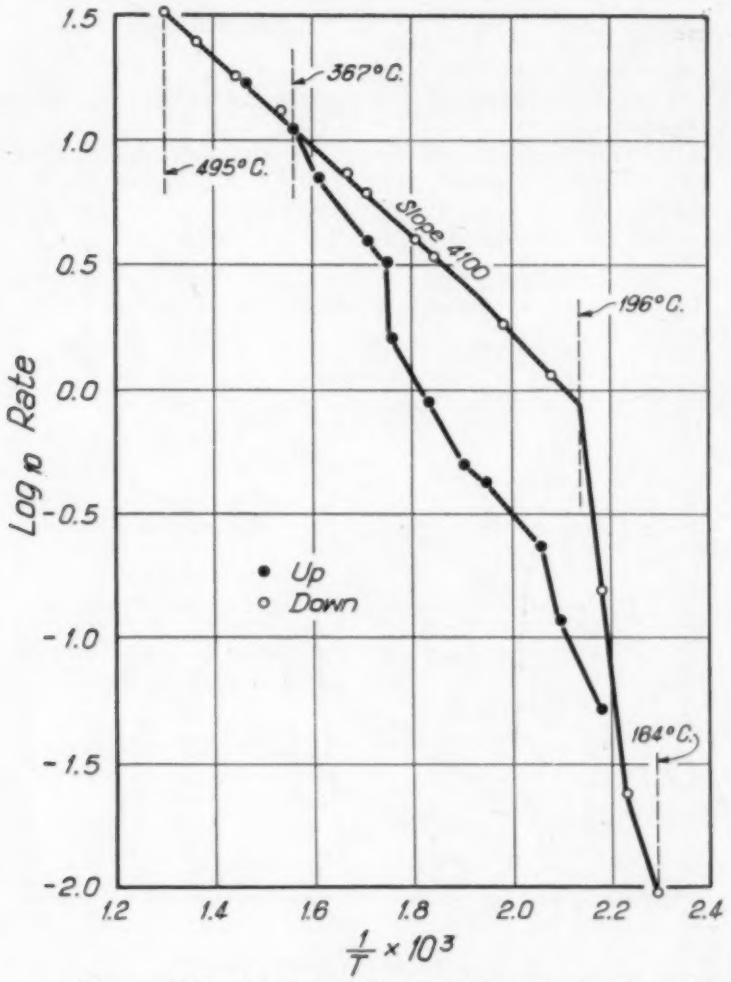

Fig. 6—Diffusion Isobar (495-164 Degrees Cent.) at Atmospheric Pressure for "Yensen" Iron—"Trapped" Method.

As previously indicated by Ham (loc. cit.), the amount by which the slope of the linear isotherm, or $\log_{10}$ Rate vs. $\log_{10}$ Pressure graph, differs from one-half can be used as a rough measure of the amount of carbon in solution in the iron sample, provided other impurities are absent. Fig. 5 shows the 500 degrees Cent. (930 degrees Fahr.) (approx.) isotherms for all samples except the one of Yensen iron. Reference to Table I clearly indicates that the isothermal slope decreased as carbon was removed by baking in

hydrogen at temperatures in excess of 1000 degrees Cent. (1830 degrees Fahr.)—decarburization at 900 degrees Cent. (1650 degrees Fahr.) is very slow. The Samples 2, 3, 4 and 5 are seen to be numbered in order of decreasing carbon content.

Figs. 6 to 14 inclusive show $\log_{10}$ Rate vs. $1/T^{\circ}K$ graphs at atmospheric pressure for Samples 1 to 5 inclusive. Fig. 6 shows the

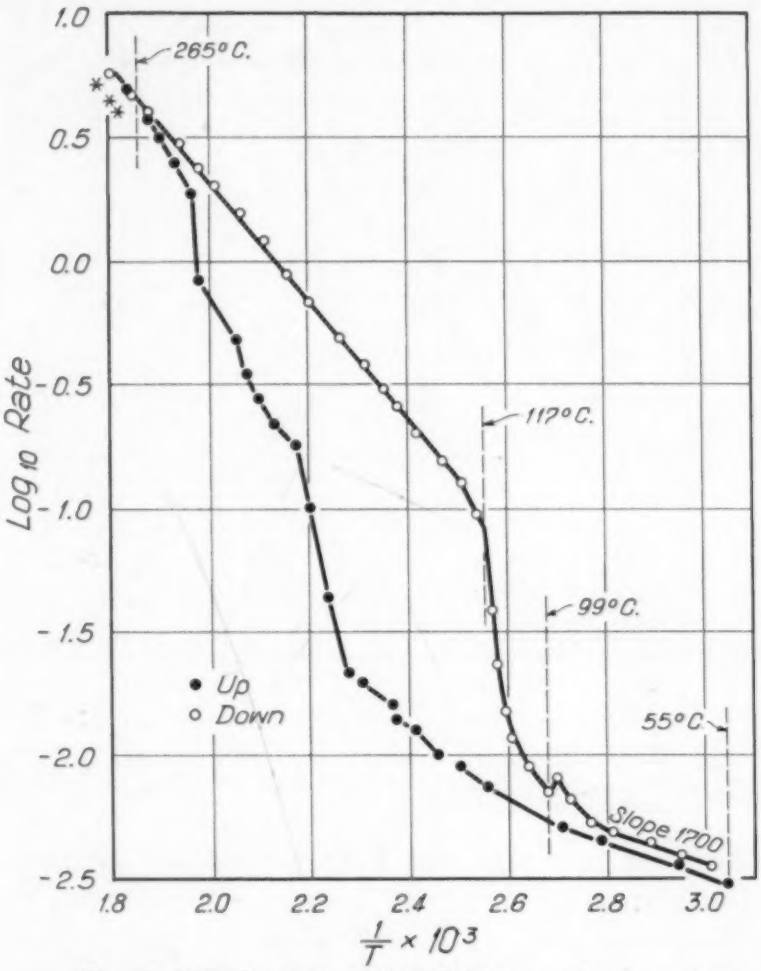

Fig. 7—Diffusion Isobar (265-55 Degrees Cent.) at Atmospheric Pressure for Nondecarburized Carbonyl Iron—"Constant Flow" Method.

low temperature isobar for Yensen iron. The high temperature isobar for this type of iron was formerly published by Ham. Fig. 7 is a detailed repetition of the low temperature region of the isobar for Sample No. 2 (see curve D, Fig. 4). The entire isobar for Sample No. 3, carbonyl iron treated 5 hours in hydrogen, is given in Figs. 8, 9 and 10, overlapping points being indicated by asterisks. For brevity the high temperature section of the isobar for Sample No. 4, carbonyl iron heated 8 hours in hydrogen, is omitted; but the

low temperature section is shown in Fig. 11. The lower section of the isobar for Sample No. 5, Mehl iron, treated 250 hours in hydrogen, is shown in Fig. 12. The magnetic transition and the beta gamma transformation are shown for this sample on separate enlarged plots in Figs. 13 and 14. Unfortunately, not more than about every third observation could be recorded on the higher temperature plots here given. A selection of typical observations is shown.

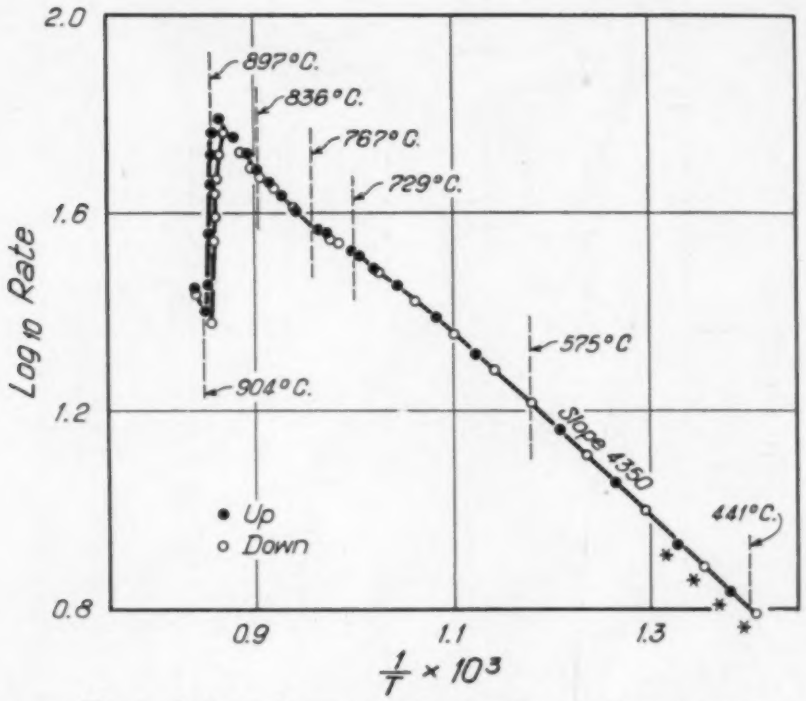

Fig. 8—Diffusion Isobar (920-441 Degrees Cent.) at Atmospheric Pressure for Carbonyl Iron Baked 5 Hours at 1100 Degrees Cent. in H_2—"Constant Flow" Method.

Interesting features of the isobars thus far presented can be listed as follows:

(a) the presence of a marked "discontinuity," accompanied by considerable hysteresis, in the low temperature range;

(b) the regularity of the low temperature sections of the isobar and the ease of duplication for decreasing temperature, as opposed to the irregularity and impossibility of exact duplication for increasing temperature;

(c) the decrease of hysteresis and shift of the low temperature discontinuity toward higher temperatures as the carbon content is decreased;

(d) the development with decrease in carbon content of a small maximum just below the low temperature discontinuity;

(e) the decrease with decrease in carbon content in the temperature spread of the low temperature discontinuity as observed going down in temperature;

(f) the increase in curvature of the isobars commencing at about 575 degrees Cent. (1070 degrees Fahr.) and extending into the

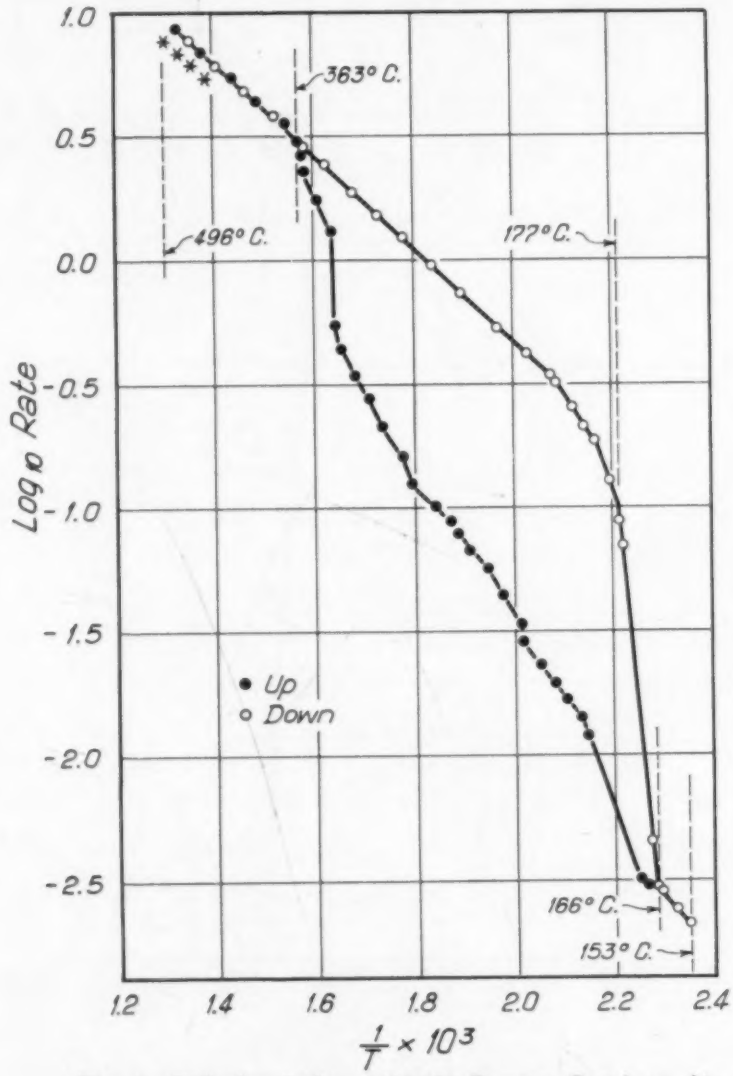

Fig. 9—Diffusion Isobar (496-153 Degrees Cent.) at Atmospheric Pressure for Carbonyl Iron Baked 5 Hours at 1100 Degrees Cent. in H_2—"Constant Flow" Method.

magnetic transition region, 730 to 770 degrees Cent. (1345 to 1420 degrees Fahr.);

(g) the independence of the magnetic transition temperatures upon change in carbon content;

(h) the increase of Ac_3 temperature with decrease in carbon content;

(i) decrease of temperature spread of the Ac_3 transformation with decrease in carbon content.

A discussion of the phenomena listed in items (a) to (e) inclusive is given later in this paper. The phenomenon mentioned

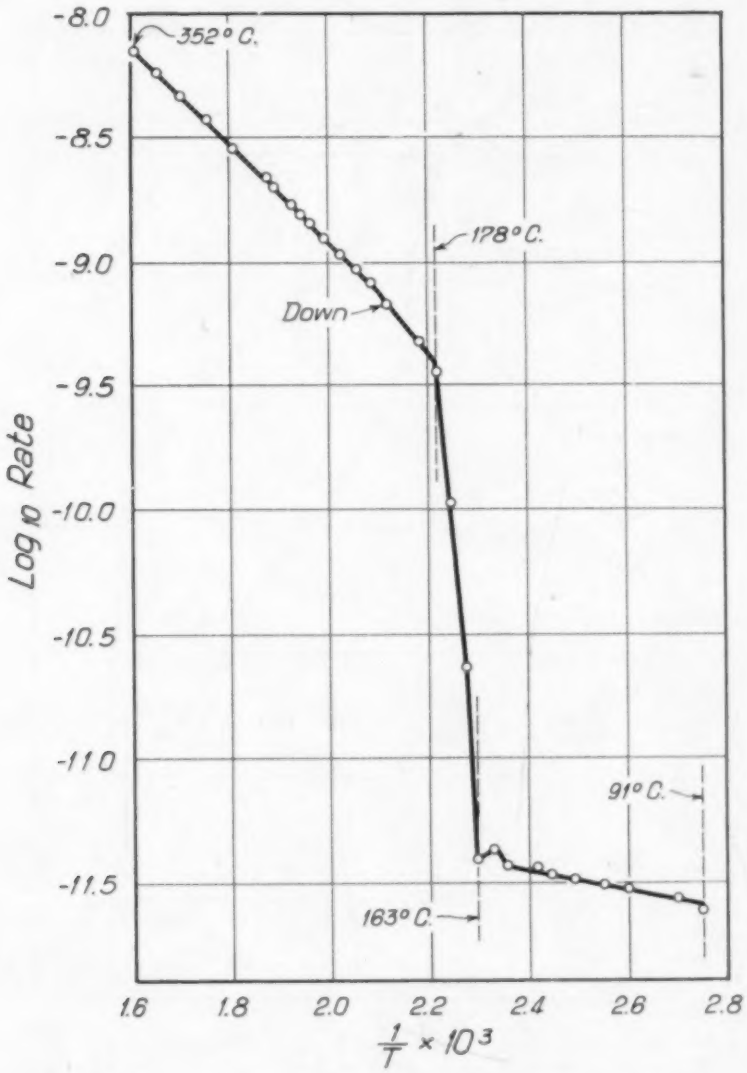

Fig. 10—Diffusion Isobar (352-91 Degrees Cent.) at Atmospheric Pressure for Carbonyl Iron Baked 5 Hours at 1100 Degrees Cent. in H_2—"Trapped" Method. Rates are Measured in Grams Per Second Through Sample. Area of Sample = 2.8 cm². Thickness of Sample = 0.155 cm.

above in item (f) can be interpreted as indicating a gradual setting-in of the magnetic transition, since the magnetism vs. temperature curve of Benedicks (5) shows a gradual curvature on the low temperature side of the A_2 region and since temperature coefficient of resistance curves show a marked change in curvature in this temperature range

(see Fig. 16). Items (g), (h) and (i) are all in agreement with accepted facts.

Important information obtained from the diffusion graphs of Figs. 4 to 14 inclusive and from additional unpublished data taken by the authors is compiled for convenience in Table II.

Table II

Sample No.	Slope 500 Degrees Cent. Isothermal[1]	Ac_3 Temp. Range Degrees Cent.	Temp. Spread of Ac_3 Degrees Cent.	Temp. of Small "Break" below Ac_3 Degrees Cent.	Magnetic Transition Temperatures Degrees Cent.	Slope Isobar 300°C. to 550°C.	Temp. Start of Ar_0 Degrees Cent.	Temp. Spread of Ar_0 Degrees Cent.
1	.....	.....	.	...	.....	4100	196	..
2	0.555	890–899	9	834	765–713	4550	117	18
3	0.550	897–904	7	836	767–729	4350	178[3]	15
4	0.535	.....	.	836	770–730	4650	238	14
5	0.520	902.0–907.5[2]	5.5	...	767–729	4450	315	12

[1]The slopes of isothermals above 700 degrees Cent. (1290 degrees Fahr.) are in all cases 0.500.

[2]C. B. Post of The Pennsylvania State College has made a careful study of the A_3 region of "Mehl" iron using the diffusion method. Reference: *Journal of Chem. Phys.*, Nov. 1937.

[3]The temperatures of Fig. 10 are used in the table, since the very low McLeod gage readings rendered inaccurate the points below the break in Fig. 9.

Fig. 15 shows isothermal graphs taken above, below, and in the midst of the low temperature discontinuity for Sample No. 4. This iron evidently contained less carbon than any other studied with the exception of that obtained from Mehl. The slopes of the isotherms increased continuously with decreasing temperature until the value 0.690 was obtained at 278 degrees Cent. (533 degrees Fahr.), just above the break in the isobar. Immediately below the break the slope attained the value 1.08. A second isotherm obtained at 278 degrees Cent. (533 degrees Fahr.), this time for increasing temperature, showed a slope of 0.830, intermediate between the slopes noted above and below the break.

The low temperature discontinuity for Sample No. 4 was examined at pressures of 74 and 42 centimeters of mercury respectively and was found to be unaltered in temperature of occurrence and in general shape.

The ease of duplication of this low temperature discontinuity is strikingly illustrated by the curves in Figs. 9 and 10. The observations of Fig. 10 were made by the "trapped method"; those in Fig. 9 were made more than two weeks later on the same sample by the "constant flow" method. In the two cases, the temperature of occurrence of the discontinuity as observed for decreasing temperature was the same within the limits of experimental error. In the par-

ticular case of the data plotted in Fig. 9, the few low temperature readings taken below the break are not accurate enough to show the small maximum present there.

The portion of the low temperature hysteresis loop associated with rising temperatures can be reproduced only approximately, but the rate of diffusion for constant temperature and pressure at any point on the hysteresis loop is independent of the time, after an initial short interval has elapsed.

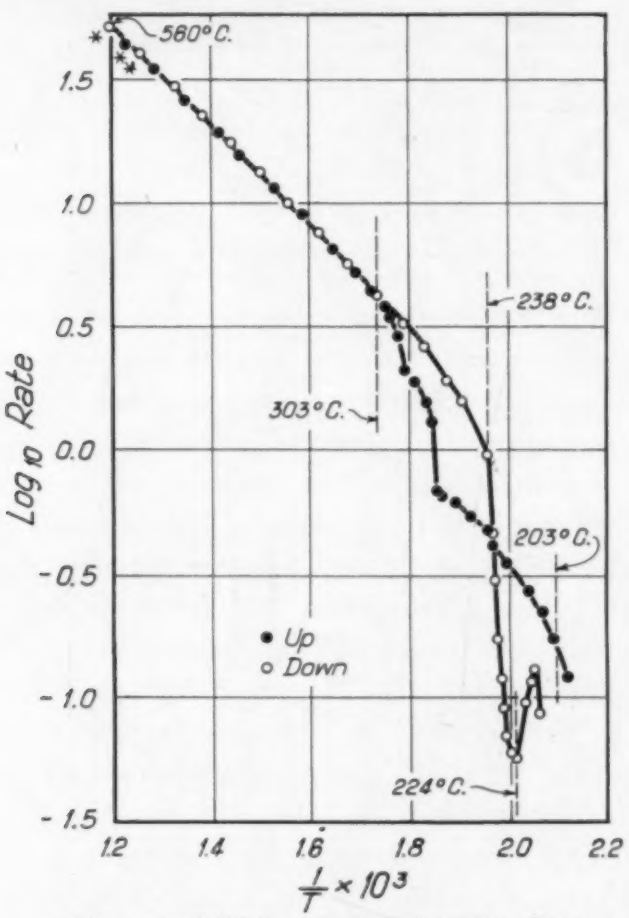

Fig. 11—Diffusion Isobar (560-203 Degrees Cent.) at Atmospheric Pressure for Carbonyl Iron Baked 8 Hours at 1100 Degrees Cent. in H_2—"Constant Flow" Method.

Discussion of Results

The experimental facts indicate that the low temperature discontinuity in the diffusion isobar is associated with a change occurring inside the iron sample. An alternative explanation, which would be in agreement with the views of Smithells and Ransley (loc. cit.), is that for a given degree of purity of sample surface there exists a

critical temperature below which activated adsorption has practically ceased. The authors do not believe that the adsorption explanation is correct. Ham (loc. cit.) has previously shown that the adsorption theory of diffusion is open to question, since the principal experimental effects which caused its inception were in reality due to con-

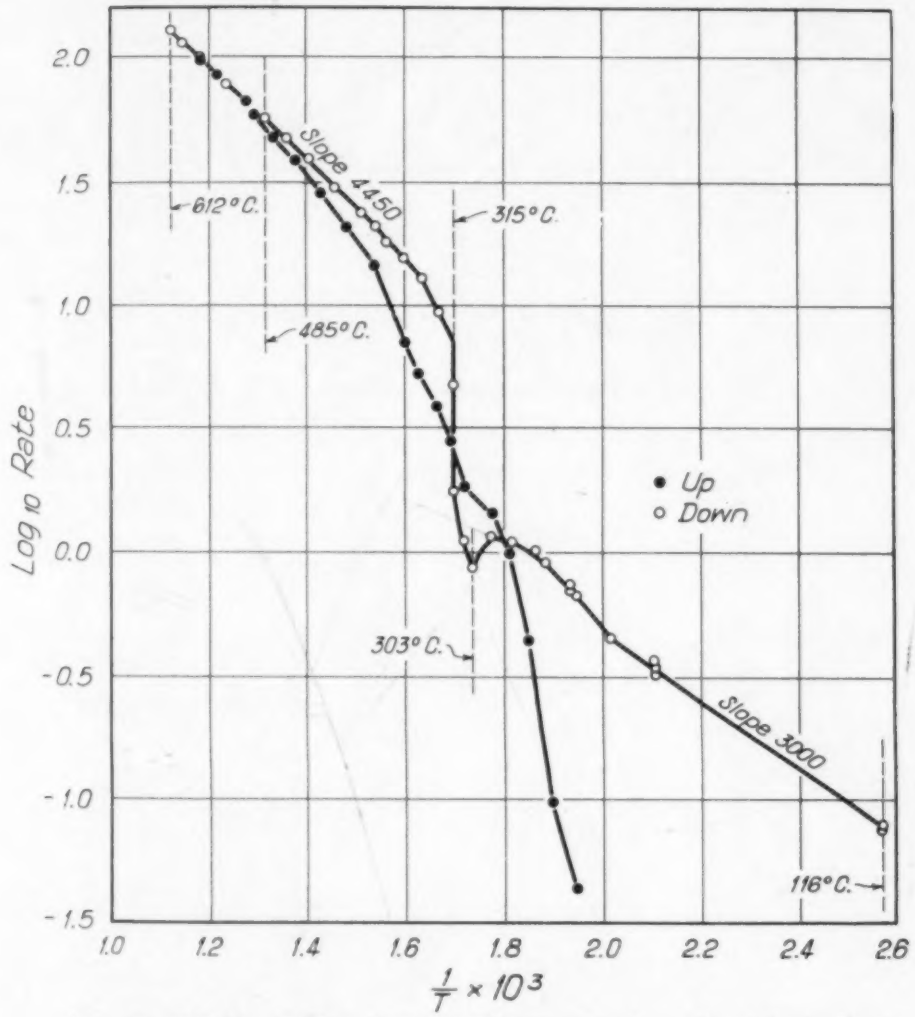

Fig. 12—Diffusion Isobar (612-116 Degrees Cent.) at Atmospheric Pressure for "Mehl" Iron—"Trapped" Method.

tamination of the metal samples used in the investigations. The linear nature of the isotherms associated with the low temperature isobaric discontinuity proves that decreasing the pressure from atmospheric to 13 centimeters of mercury does not shift the isobar along the 1/T°K scale. If the low temperature discontinuity were a critical region for adsorption, a truly linear isotherm in the midst of the erratic lower half of the hysteresis loop would be impossible, since the amount of adsorption present must depend upon the pres-

sure. The increase of isotherm slopes to unity as the low temperature discontinuity is traversed seems to indicate that in this region of the isobar the state of aggregation of the hydrogen ions or excited atoms inside the sample undergoes an abrupt change. It is suggested

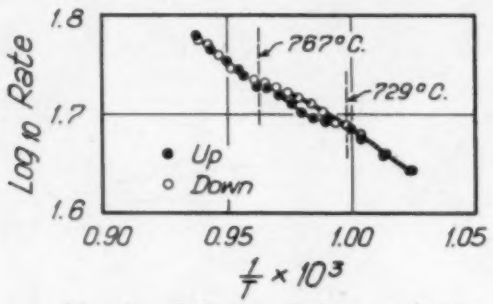

Fig. 13—Diffusion Isobar at Atmospheric Pressure for the Magnetic Transition Region of "Mehl" Iron—"Constant Flow" Method.

that at the A_0 transition a change in valence of the iron atoms inside the crystal lattice occurs. Such an electronic rearrangement would change the binding forces between the iron atoms and the hydrogen

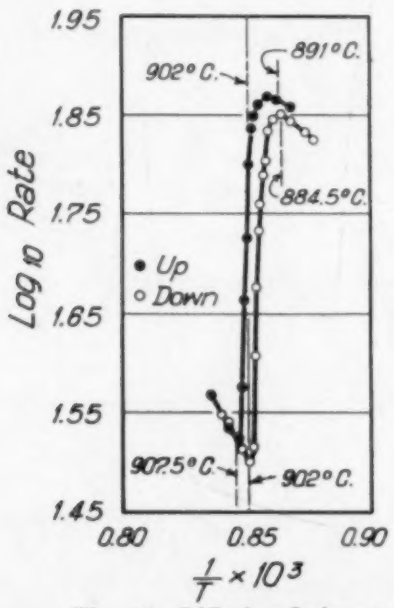

Fig. 14—Diffusion Isobar at Atmospheric Pressure for the A_2 Transformation of "Mehl" Iron —"Constant Flow" Method.

ions or excited atoms and would consequently result in a change of the state of aggregation of the hydrogen inside the metal.

As arguments that the observed low temperature discontinuity in the diffusion isobar results from a critical change in the iron can

be cited the facts that hysteresis is present; that an increase in carbon content shifts the discontinuity to a lower temperature; and that the temperature spread of the discontinuity for decreasing temperature increases with increase in carbon content. It is well known that hysteresis is associated with the A_3 and A_2 critical points of iron, that

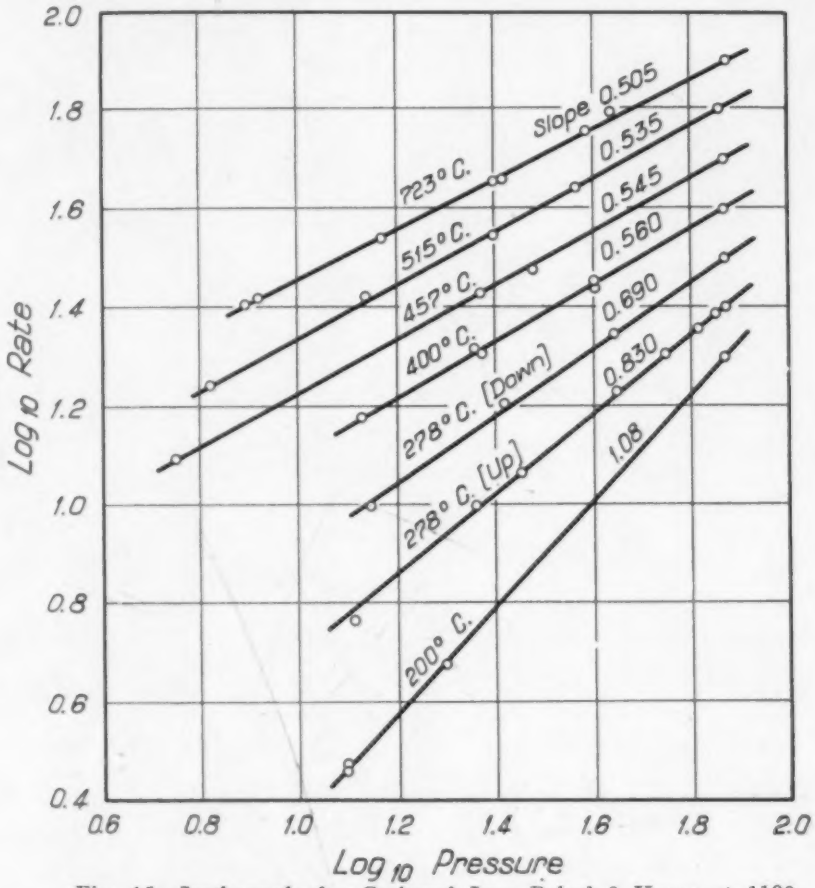

Fig. 15—Isothermals for Carbonyl Iron Baked 8 Hours at 1100 Degrees Cent. in H_2.

increase in carbon content shifts the A_3 point of iron toward lower temperatures, and that the temperature spread of the Ac_3 point of iron increases with increase of carbon content.

A pronounced maximum and minimum are to be found in the ultimate tensile strength versus temperature curve of nearly pure iron (6) in the temperature range of the diffusion discontinuity. In Fig. 4 the curve T, ($\log_{10}$ 1/U.T.S. vs. 1/T°K) for electrolytic iron is compared with the curve D, ($\log_{10}$ Rate vs. 1/T°K) for non-decarburized carbonyl iron. Note the similarity in appearance of the curves at higher temperatures. This leads one to believe that, at least for high temperatures, the rate of diffusion of hydrogen

through the iron varies in some inverse manner with the binding forces between the lattice points of the iron.

A small discontinuity in the temperature coefficient of resistance vs. 1/T°K graph of Fig. 16 appears at about 270 degrees Cent. (520 degrees Fahr.). The degree of purity of this iron is not known accurately, but the fact that the beta-gamma transition occurs at a

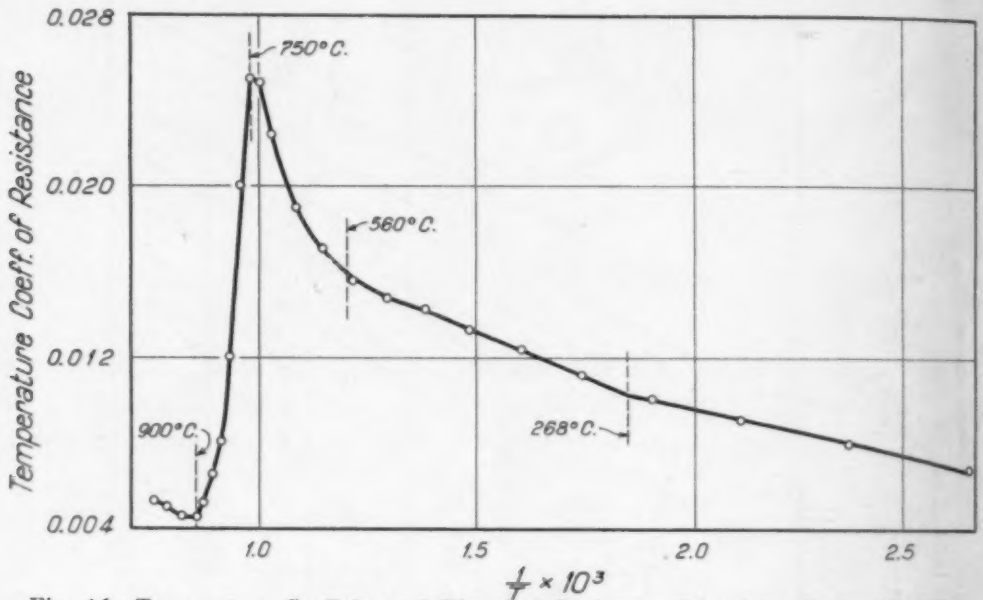

Fig. 16—Temperature Coefficient of Electrical Resistance Plot from Somerville (7).

temperature of about 900 degrees Cent. (1650 degrees Fahr.) indicates that the sample is only slightly contaminated with carbon.

In consequence of the data herein presented, it appears that the low temperature discontinuity noted in the diffusion isobars is ascribable to a thermal critical point in iron of very low carbon content. Furthermore, it seems probable that complete decarburization will not remove the discontinuity noted and that an A_0 point for pure iron exists.

References

1. W. R. Ham, TRANSACTIONS, American Society for Metals, Vol. 25, 1937, p. 536.
2. Borelius and S. Lindblom, *Ann. d. Physik*, Vol. 82, 1927, p. 201.
3. C. J. Smithells and C. E. Ransley, *Proceedings*, Royal Society, Vol. 150, 1935, p. 172.
4. C. Wells, R. A. Ackley and R. F. Mehl, "A Dilatometric Study of the Alpha-Gamma Transformation in High Purity Iron," TRANSACTIONS, American Society for Metals, Vol. 24, 1936, p. 46.
5. Benedicks—See Sauveur, "The Metallography and Heat Treatment of Iron and Steel," p. 131.
6. See Sauveur (loc. cit.), p. 137.
7. A. A. Somerville, *Physical Review*, Vol. 31, 1910.

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